

METHOD FOR PRODUCTION OF GLYCOLIDE

BACKGROUND OF THE INVENTION

This invention pertains to a method for continuously producing high-purity glycolide from glycolic acid oligomers or methyl glycolate oligomers. The process includes generating crude glycolide by depolymerizing these oligomers, followed by refining the crude product through melt crystallization to achieve ultrapure glycolide.

Polyglycolic acid (PGA) is a biodegradable, biocompatible polymer renowned for its excellent mechanical properties, gas barrier characteristics, and hydrolyzability. Initially developed for medical applications like sutures and tissue engineering, PGA's use has expanded to packaging, industrial products, and environmental applications due to its eco-friendly nature. Its natural degradability makes it an appealing choice for reducing environmental impact.

Glycolide is a cyclic dimer of glycolic acid and serves as an intermediate in the production of high molecular weight PGA. The production process involves a technique known as ring-opening polymerization. During this process, glycolide molecules are subjected to heat and catalysis, causing the cyclic dimer ring structure to break. This action initiates a chain reaction in which the glycolide monomers link together, forming long polymer chains that constitute high molecular weight PGA.

One established method for synthesizing glycolide involves a pre-polymerization step followed by a depolymerization step. In this process, glycolic acid undergoes a reaction that removes water as a byproduct, resulting in the formation of glycolic acid oligomers. These oligomers are then depolymerized to produce crude glycolide. Alternatively, alkyl glycolate such as methyl glycolate can be used as the starting material. In this approach, pre-polymerization occurs through the removal of alcohol (dealcoholization), leading to the formation of alkyl glycolate oligomers, with alcohol as the byproduct. These oligomers are then depolymerized in a similar way to yield crude glycolide.

Various processes have been proposed for preparing glycolide from the generated glycolic acid oligomers. U.S. Patent No. 9,512,100, for example, describes a method where glycolic acid oligomers are heated with a large amount of tetraethylene glycol dibutyl ether as a polar organic solvent and octyl triethylene glycol as a solubilizing agent during the depolymerization process. In this method, the crude glycolide formed is co-distilled with tetraethylene glycol dibutyl ether. The resulting co-distillation product then undergoes phase separation into a glycolide-enriched phase and a polar organic solvent-enriched phase. The crude glycolide can be obtained by isolating and recovering the glycolide from the glycolide-enriched phase in its liquid form.

While the method described in U.S. Patent No. 9,512,100 allows for obtaining crude glycolide from glycolic acid oligomers using a polar organic solvent and solubilizing agent, it presents several disadvantages. Firstly, the process requires a large volume of polar organic solvent for the co-distillation with crude glycolide, leading to significant energy consumption due to the need to boil the solvent. Secondly, the phase separation of the co-distillation product into a glycolide-enriched phase and a polar organic solvent-enriched phase followed by recovering crude glycolide from the glycolide-enriched phase necessitates additional operational steps, adding complexity to the process. Moreover, some of the organic solvents or solubilizing agents cited in U.S. Patent No. 9,512,100 are known to be toxic, and certain substances are prone to decomposition, rendering them unfit for repeated use.

SUMMARY OF THE INVENTION

It is therefore an object of the present invention to develop a method for producing crude glycolide through the depolymerization of glycolic acid oligomers or alkyl glycolate oligomers. This method involves introducing a small quantity of liquid substance, as part of the reaction feed, into the depolymerization reactor during the depolymerization process. This liquid substance readily dissolves in the oligomers to form a homogeneous solution with reduced viscosity, and acts as a stripping agent to facilitate the efficient generation of crude glycolide from the oligomers.

It is another object of the present invention to provide a method for producing highly pure glycolide from the obtained crude glycolide by suspension-based melt crystallization, while the mother liquor from the crystallization process is fed back into the pre-polymerization system.

BRIEF DESCRIPTION OF THE DRAWING

FIG.1 is a schematic representation of a preferred glycolide production system using glycolic acid as the starting material in accordance with the present invention.

FIG.2 is a schematic representation of a preferred glycolide production system using methyl glycolate as the starting material in accordance with the present invention.

FIG.3 is an alternative schematic representation of a preferred glycolide production system using glycolic acid as the starting material in accordance with the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

Two common approaches for synthesizing oligomers involve pre-polymerizing glycolic acid or pre-polymerizing via dealcoholization using alkyl glycolate as the starting material. In the first approach, glycolic acid undergoes a condensation reaction to generate glycolic acid oligomers,

with water formed as a byproduct and subsequently removed. In the second approach, dealcoholization converts alkyl glycolate into alkyl glycolate oligomers by removing the alcohol group. For instance, methyl glycolate undergoes dealcoholization to form methyl glycolate oligomers and methanol, while ethyl glycolate undergoes dealcoholization to produce ethyl glycolate oligomers and ethanol.

Glycolic acid and alkyl glycolates, particularly methyl glycolate, are both commercially available and commonly used in the production of glycolide. Glycolic acid can also be synthesized from methyl glycolate through a hydrolysis process, where the ester bond in methyl glycolate is broken down with water, often assisted by a catalyst, resulting in the formation of glycolic acid and methanol.

In glycolide production, choosing between glycolic acid and methyl glycolate requires balancing factors such as ease of handling, reactivity, and supply considerations. Glycolic acid, being more reactive during the pre-polymerization step, is an efficient starting material for producing glycolic acid oligomers. However, its high melting point of 80°C requires careful handling to prevent freezing in equipment such as vacuum systems, pipes, and fittings. Conversely, methyl glycolate has a significantly lower melting point, making it easier to handle and process, but it is less reactive than glycolic acid. Therefore, the choice between glycolic acid and methyl glycolate in glycolide production depends on these trade-offs between reactivity and handling convenience.

Glycolic acid, with its simpler molecular structure and lack of a chiral center, does not have optical isomers. This simplicity provides glycolic acid and its derivative, methyl glycolate, with inherent stability, preventing racemization even under prolonged high temperatures. This stability allows for the production of high molecular weight oligomers without the risk of racemization. In contrast, lactic acid, which has two optical isomers due to its chiral center, is susceptible to racemization at elevated temperatures. This racemization can result in the formation of undesired meso-lactide during the depolymerization of lactic acid oligomers, even when starting with a pure enantiomer of lactic acid.

During the depolymerization of glycolic acid or methyl glycolate oligomers, intramolecular reactions occur in which the end of the polymer chain "bites" back to form a cyclic dimer, resulting in the production of glycolide. The efficiency of this backbiting process is significantly influenced by the chain length of the oligomers, which corresponds to the number of carbon atoms.

Longer oligomer chains can enhance the intramolecular reactions necessary for backbiting, which promotes increased glycolide formation. However, as the chains become too long, the viscosity of the oligomers rises. This presents a challenge because glycolide, with its significantly higher boiling point compared to lactide, becomes more difficult to distill from the viscous oligomers.

Moreover, glycolide is much more reactive than lactide and, if not promptly removed, can rapidly repolymerize due to the reversible nature of oligomer depolymerization. This repolymerization results in the formation of more viscous oligomers, leading to longer residence

times in the depolymerization reactor. The extended exposure to elevated temperatures increases the risk of degradation and tar formation, ultimately reducing the overall yield of glycolide production. One approach to address these challenges, as described in U.S. Patent No. 9,512,100, involves the use of a large quantity of polar organic solvent and solubilizing agent during depolymerization. Although this method is effective, it also requires a significant amount of energy to evaporate the co-distilled solvent, thereby adding complexity and increasing the cost of the process.

On the other hand, if the chains are too short, their backbiting efficiency decreases, leading to reduced glycolide formation. As a result, these short-chain oligomers accumulate in the depolymerization reactor, causing the level in the pre-polymerization reactor to rise and increasing residence times due to their inefficient conversion into glycolide. This, in turn, heightens the risk of degradation and tar formation.

The inventors have found that glycolic acid or methyl glycolate oligomers, with an average number molecular weight ranging from 800 to 2,000, exhibit satisfactory backbiting efficiency. These oligomers predominantly consist of chains with a degree of polymerization between 10 and 40, making them an ideal feedstock for subsequent depolymerization reactions. However, these oligomers still have a relatively high viscosity, making it difficult to effectively remove the generated glycolide. To address this issue, a small amount of liquid substance can be introduced into the depolymerization reactor as part of the reaction feed. This substance, which blends well with the oligomers, helps to lower their viscosity and functions as a stripping agent, thereby improving both the formation and extraction of glycolide.

The production of glycolide typically begins with an aqueous solution composed of 70% glycolic acid and 30% water by weight. The first step in the production process is pre-polymerization, which involves a first reactive distillation process. During this step, the concentration of glycolic acid is increased, leading to a condensation reaction that produces glycolic acid oligomers and water as a byproduct. This pre-polymerization can be performed in a single stage or multiple stages, such as two stages connected in series, particularly when processing large quantities of feedstock.

After the formation of glycolic acid oligomers, they undergo a second reactive distillation process, during which the oligomers are depolymerized to produce crude glycolide. The inventors have found that introducing a small quantity of concentrated glycolic acid, preferably with a concentration of over 95% glycolic acid by weight, into the depolymerization reactor can significantly boost glycolide production.

The concentrated glycolic acid is favored over the initial glycolic acid solution, which contains 30% water by weight. The presence of water in the initial solution can lead to increased water vapor formation when introduced into the hot depolymerization feed. Excessive water vapor in the depolymerization reactor reduces the heat transfer coefficient on the process flow side, leading to a decrease in the reactor's overall heat transfer efficiency. Additionally, water vapor is challenging to condense, potentially carrying other substances such as glycolic acid into the vacuum system and complicating the process further.

The addition of concentrated glycolic acid solution provides several key advantages. Firstly, it dissolves efficiently in the oligomers, resulting in a homogeneous solution and significantly lowering the mixture's viscosity. This reduction in viscosity allows the generated glycolide to escape more easily from the oligomers. Secondly, with the residence time in the depolymerization reactor being less than three minutes, only a portion of the added glycolic acid has the opportunity to form glycolic acid dimers or react with the oligomers, both of which produce water as a byproduct. These formed dimers, along with the water and unreacted glycolic acid, have much lower boiling points than glycolide, enabling them to function as stripping agents that assist in the removal of glycolide from the oligomers. Lastly, since the added glycolic acid makes up only a small fraction of the depolymerization feed, the subsequent glycolide purification process becomes simpler and requires significantly less energy.

In the first reactive distillation, the glycolic acid concentration and glycolic acid condensation reaction are allowed to proceed for the generation of glycolic acid oligomers. The average number molecular weight of the glycolic acid oligomers obtained as a result of the glycolic acid condensation reaction generally ranges from 400 to 5,000, preferably ranges from 600 to 3,000, and more preferably ranges from 800 to 2,000.

In accordance with the present invention, the first reactive distillation system preferably includes at least a pot, a distillation column, a condenser, and an evaporator. In this system, the distillation column, with the condenser mounted horizontally on top, and the evaporator are preferably respectively mounted on top of the pot. This arrangement creates a single enclosed space where glycolic acid concentration, condensation, and the associated distillation processes take place.

The orientation of the pot can be horizontal or vertical depending on the process conditions. The distillation column can be a conventional column or a divided wall column with a divided wall dividing the inner space of column. The present invention is not particularly limited with regard to the type of mass transfer elements installed in the distillation column. Good results are obtained by using suitable mass transfer elements selected from the group consisting of trays, random packings, structured packings and any combinations thereof. It is however, structured packings are particularly suitable as mass transfer elements with the advantages of reducing the column pressure drop and liquid hold-up in the column. It is preferred that the structured packings have a specific surface area in the range of 50 to 750 m²/m³, and more preferably of in the range of 125 to 500 m²/m³. The evaporator can be of any of the types commonly found in the chemical industry, including, but not limited to, falling film, forced circulation, thermosiphon and etc. However, due to its particularly reduced liquid hold-up, a falling film evaporator is preferred to decrease the residence time of glycolic acid and oligomers and therefore reduce any unfavorable side-reactions, for example degradation and tar formation. The condenser can be of any of the types commonly used in the chemical industry including co-current and counter-current condensers. However, a modified horizontal shell and tube heat exchanger as described in WO2022/177500 is preferred, which is mounted on the top of the distillation column. Due to its specific geometric configuration, this condenser causes only a very small pressure drop. This small pressure drop translates into lower operating pressures for the evaporator and the pot,

which in turn leads to a lower condensation reaction temperature in the evaporator, thereby minimizing any unfavorable side reactions, such as degradation and tar formation.

The initial glycolic acid solution is continuously fed into the distillation column through an inlet positioned between the upper and lower ends of the column. The reaction feed enters at the top of the falling film evaporator, flowing down through long vertical tubes that serve as the heat transfer and reaction zone. These tubes are surrounded by a heat transfer medium, such as heating oil or steam, circulating on the shell side. As the reaction solution passes through the tubes, it exits at the bottom as a vapor-liquid two-phase mixture. This two-phase stream flows directly into the connected pot, where the vapor is separated from the liquid. The separated vapor rises to the bottom of the top-mounted distillation column, while the liquid is collected in the pot. To prevent the breakdown of the liquid film inside the tubes of the falling film evaporator, the vaporization of the reaction solution is typically kept below 30% by weight. A significant portion of the liquid collected at the bottom of the pot is recirculated via a transfer pump back to the top of the falling film evaporator for continuous glycolic acid condensation. A smaller portion of the liquid is fed into the subsequent depolymerization reactor. The reaction solution is heated in general at temperatures ranging from 120 to 220°C. and preferably ranging from 160 to 200°C.

In the distillation column of the first reactive distillation system, a concentration gradient is established, with water becoming concentrated in the rectifying section and higher boiling point components, such as glycolic acid and its oligomers, concentrating in the stripping section. The water present in the aqueous glycolic acid stream, along with the water generated during glycolic acid condensation, is distilled off as an overhead vapor stream. This vapor is condensed by the top-mounted modified horizontal condenser, producing a condensate stream composed primarily of water. A portion of this condensate is preferably refluxed back into the column, while the remaining portion can be discarded. Any vapors not condensed are removed by the vacuum system. The high-boiling fraction, mainly consisting of glycolic acid and its oligomers, liquefies within the column and flows back into the pot.

In accordance with the present invention, the depolymerization of glycolic acid oligomers is conducted within a second reactive distillation system, which preferably includes at least a pot, a distillation column, a condenser, and a falling film evaporator. The distillation column and the falling film evaporator are directly mounted on top of the pot, forming a single enclosed space where both depolymerization and distillation occur. The mass transfer elements installed in the distillation column consist of trays, random packings, structured packings and any combinations thereof. Structured packings are particularly advantageous due to their ability to reduce pressure drops and minimize liquid hold-up within the column. It is preferred that the structured packings have a specific surface area ranging from 50 to 750 m²/m³, with a preferred range of 125 to 500 m²/m³. The falling film evaporator is chosen for this process because it significantly reduces liquid hold-up, thereby decreasing the residence time of glycolic acid oligomers. This reduction in residence time is crucial for minimizing unfavorable side reactions, such as degradation and tar formation. The system also incorporates a modified horizontal shell and tube heat exchanger, as detailed in WO2022/177500, which serves as the condenser and is mounted on top of the distillation column. This condenser's unique geometric design results in a very small pressure

drop, which in turn lowers the operating pressures for both the evaporator and the pot. This reduction in pressure leads to a lower depolymerization reaction temperature within the evaporator, further minimizing the risk of undesirable side reactions like degradation and tar formation.

A small portion of the initial glycolic acid solution, composed of 70% glycolic acid and 30% water by weight, is first directed into a separate distillation column where the glycolic acid concentration is preferably increased to over 95% by weight. The concentrated glycolic acid stream is then introduced and mixed with the glycolic acid oligomers from the first reactive distillation system. The concentrated glycolic acid solution content in the glycolic acid oligomers stream from the first reactive distillation system is preferably between 1% and 10% by weight, with a more preferred range of 3% to 6% by weight.

In the second reactive distillation system, a catalyst such as tin dioctoate is combined with the glycolic acid oligomers from the first reactive distillation system, along with the small amount of concentrated glycolic acid solution that is introduced. This mixture is then fed into the recirculation stream of the falling film evaporator, forming the reaction feed. In this setup, the falling film evaporator functions as both the depolymerization reactor, where glycolic acid oligomers are converted into crude glycolide, and the energy source for evaporating the resulting glycolide and other low-boiling volatiles, such as glycolic acid dimers, unreacted glycolic acid, and water.

The reaction feed enters the top of the falling film evaporator and flows downward through long vertical tubes, which are heated by a medium, such as heating oil or steam, circulating on the shell side. As the reaction progresses, the solution exits at the bottom of the tubes in a vapor-liquid two-phase form.

The vapor-liquid two-phase stream exits at the bottom of the tubes in the falling film evaporator and flows directly into the connected pot, where the vapor is separated from the liquid. The disengaged vapor then rises to the bottom of the distillation column mounted above the pot, while the separated liquid is collected in the pot. To prevent the breakdown of the liquid film inside the tubes of the falling film evaporator, the vaporization of the reaction solution is typically kept below 30% by weight.

A significant portion of the liquid collected at the bottom of the pot is recirculated via a transfer pump back to the top of the falling film evaporator for continuous depolymerization. A small portion of this liquid is removed as a purge stream, which contains residues of the tin catalyst and any metals leached from the system.

The operating pressure of the horizontal condenser is set within a range of 3 to 30 mbar, with a more preferred range of 5 to 10 mbar. The reaction solution is heated in the falling film evaporator at temperatures preferably ranging from 200 to 280° C, and more preferably ranging from 220 to 240°C. An overhead low-boiling distillate stream, known as crude glycolide, is generated. This stream primarily consists of 75 to 97% by weight glycolide, along with smaller amounts of glycolic acid dimers (0 to 10% by weight), unreacted glycolic acid (0 to 8% by weight), water (0 to 6% by weight), and glycolic acid oligomers (0 to 1% by weight). The high-

boiling fraction, which consists mainly of unconverted glycolic acid oligomers, flows back into the pot for further processing.

As the demand for ultrapure glycolide with a purity of 99.9% or higher increases in the medical industry, further purification of crude glycolide is essential to achieve the desired purity. However, due to its high reactivity, purifying crude glycolide in a distillation system poses a risk of repolymerization into oligomers, especially at the bottom of the distillation column where temperatures are higher. In contrast, melt crystallization has emerged as an optimal solution. This method operates at lower temperatures and offers a highly selective separation process that is both energy-efficient and environmentally friendly, requiring no additional solvents. Melt crystallization is divided into two categories: suspension-based melt crystallization and layer crystallization, depending on the technique used to obtain the crystalline product. While both techniques are effective for purification, suspension-based melt crystallization is generally preferred for achieving the highest purity levels, though layer crystallization remains a viable option.

To achieve high-purity glycolide, the crystallization system typically includes at least one primary stage of suspension-based melt crystallization to purify crude glycolide obtained from the depolymerization step. This suspension-based melt crystallization system features a single scraped surface drum crystallizer and at least one wash column, which together ensure the efficient separation of glycolide crystals from the impurity-enriched mother liquor, resulting in an ultrapure liquid glycolide product.

The crude glycolide enters the scraped surface drum crystallizer, where it is cooled by a heat transfer fluid circulating through the outer jacket. This cooling process forms a supersaturated film of glycolide on the inner wall of the crystallizer. To prevent encrustation and maintain efficient continuous operation, the inner wall is regularly scraped, ensuring the cooling surface stays clear. The sub-cooled liquid is then quickly mixed with the crystallizer's volume, allowing the supersaturation to disperse throughout the slurry, establishing a stable environment for controlled crystal growth. The resulting slurry is then directed to a wash column for the complete separation of ultrapure glycolide crystals from the impurity-rich mother liquor.

In the wash column, most of the impurity-rich mother liquor is removed from the glycolide crystals through filtration or other means. A large portion of this mother liquor is recycled to be combined with the initial glycolic acid solution, while a smaller portion is removed as a purge stream to prevent impurity buildup in the system. The crystals, now largely free of mother liquor, form a compacted crystal bed. While the crystals themselves are ultrapure glycolide, a thin film of residual mother liquor may remain on their surfaces. To address this, a portion of the ultrapure glycolide crystals is cut from the crystal bed and melted using steam or another appropriate heat source. A small amount of this melted glycolide is then pressed through the crystal bed to wash away the adhering mother liquor, while the majority of the melted glycolide is discharged as the final product, with a purity of 99.9% or greater, via an automated control valve.

To enhance the yield of glycolide product, a secondary stage of suspension-based melt crystallization is implemented downstream of the primary stage. In this secondary stage,

glycolide is recovered from the impurity-rich mother liquor that was separated during the primary stage. The recovered glycolide is then combined with the crude glycolide from the depolymerization step and reintroduced into the scraped surface drum crystallizer in the primary stage. Meanwhile, the impurity-rich mother liquor from the secondary stage is split into a purge stream and a recycle stream, with the latter being combined with the initial glycolic acid solution. The combined yield of glycolide from both stages of suspension-based melt crystallization exceeds 80%.

The uncondensed vapors from the top-mounted horizontal condenser of the second reactive distillation column primarily consist of water, with smaller amounts of leaked air, unreacted glycolic acid, and glycolic acid dimers. These vapors are routed to a scrubber, where a cold stream of concentrated methyl glycolate solution is introduced at the top. The purpose of this stream is to absorb the unreacted glycolic acid and dimers. Methyl glycolate, with its very low freezing point, can be sufficiently cooled to effectively condense and absorb these components, thus preventing blockages in the vacuum system and associated piping. The vapors that are not recovered at the top of the scrubber, which are mainly composed of methyl glycolate, leaked air, and water, are then directed to a vacuum system. The liquid mixture that collects at the bottom of the scrubber is recycled by combining it with the initial glycolic acid solution, which is fed into the pre-polymerization reaction in the first reactive distillation system. This recycling is feasible because methyl glycolate can also act as a starting material for glycolide production. Additionally, the uncondensed vapors from the first reactive distillation column, which contain a large portion of methanol, are directed to a vacuum system for recovery.

Similarly, to produce ultrapure glycolide starting from concentrated methyl glycolate solution, the process involves two reactive distillation systems followed by at least one stage of suspension-based melt crystallization. In the first reactive distillation system, methyl glycolate is pre-polymerized into methyl glycolate oligomers. These oligomers are then fed into the second reactive distillation system, where they are depolymerized to produce crude glycolide.

It has been found that adding a small amount of concentrated methyl glycolate solution, preferably with a concentration of over 95% by weight, to the methyl glycolate oligomers from the first reactive distillation stream significantly enhances glycolide production during the depolymerization step. In the second reactive distillation system, a catalyst such as tin dioctoate is combined with the methyl glycolate oligomers from the first reactive distillation system, along with the small amount of concentrated methyl glycolate solution that is introduced. This mixture is then introduced into the recirculation stream of the falling film evaporator, forming the reaction feed. In this setup, the falling film evaporator functions as both the depolymerization reactor, where methyl glycolate oligomers are converted into crude glycolide, and the energy source for evaporating the resulting glycolide and other low-boiling volatiles, such as methyl glycolate dimers, unreacted methyl glycolate, and methanol.

The concentrated methyl glycolate solution content in the methyl glycolate oligomers stream from the first reactive distillation system is preferably between 1% and 10% by weight, with a more preferred range of 3% to 6% by weight. The operating pressure of the horizontal condenser

in the second reactive distillation system is set within a range of 3 to 30 mbar, with a more preferred range of 8 to 15 mbar.

This addition of methyl glycolate offers several key benefits. Firstly, methyl glycolate dissolves efficiently in the oligomers, creating a homogeneous solution and greatly reducing the mixture's viscosity. The lowered viscosity allows the crude glycolide to escape easily from the oligomers. Secondly, with a residence time of less than three minutes in the depolymerization reactor, and given that methyl glycolate is less reactive than glycolic acid, only a small fraction of the added methyl glycolate forms dimers or reacts with the oligomers, generating methanol as a byproduct. The dimers, along with methanol and unreacted methyl glycolate, have much lower boiling points than glycolide, enabling them to act as stripping agents that help remove glycolide from the oligomers. Finally, because the added methyl glycolate constitutes only a small portion of the depolymerization feed, the subsequent purification process for glycolide becomes much simpler and requires considerably less energy.

Since methyl glycolate has a much lower freezing point than glycolic acid and is also less reactive, the process using methyl glycolate as the starting material differs from the process that uses glycolic acid in several ways. Firstly, methyl glycolate is polymerized into oligomers in the presence of a catalyst, which may include a tin salt, zinc salt, titanium salt, or a combination of these. Secondly, unlike the process that uses glycolic acid as the starting material, a scrubber is not needed when using methyl glycolate due to its very low freezing point. This lower freezing point prevents the risk of blockage in the vacuum system and connected pipes. As a result, uncondensed vapors from the horizontally top-mounted condenser of the second reactive distillation column can be further condensed in a cold trap. The condensate from the bottom of the cold trap contains a large portion of methyl glycolate and is then recycled back into the pre-polymerization step. The remaining uncondensed vapors, primarily composed of methanol along with methyl glycolate, leaked air and water, are directed into a vacuum system.

Adding a small amount of concentrated glycolic acid or methyl glycolate solution to the oligomers from the first reactive distillation has been observed to serve a similar dual purpose: both reduce the viscosity of the mixture and act as stripping agents during the depolymerization step. Therefore, when glycolic acid solution is the starting material, either a concentrated methyl glycolate solution alone or a combination of concentrated methyl glycolate and glycolic acid solutions can be introduced as substitutes for the concentrated glycolic acid solution in the reaction feed during the depolymerization step.

Conversely, when methyl glycolate solution is the starting material, a concentrated glycolic acid solution alone or a mixture of concentrated methyl glycolate and glycolic acid solutions can be introduced as substitutes for concentrated methyl glycolate solution in the reaction feed during the depolymerization step. However, when using glycolic acid solution, either alone or in combination with other substances, it is generally necessary to replace the cold trap with a scrubber. This adjustment is needed due to glycolic acid's significantly higher freezing point compared to methyl glycolate, requiring the absorption of uncondensed glycolic acid vapors from the condenser of the second reactive distillation column to prevent freezing in the vacuum system.

FIG.1 illustrates a preferred system for glycolide production system using glycolic acid solution as the starting material in accordance with the present invention. The system includes a first reactive distillation column 6, a condenser 7, a pot 12, a falling film evaporator 13, a pump 15, a second reactive distillation column 27, a condenser 28, a pot 20, a falling film evaporator 26, a pump 22, a scrubber 32, a concentration distillation column 41 and a suspension-based melt crystallization system 46.

An aqueous solution of glycolic acid is continuously introduced via stream 1 into a static mixer 2, where it combines with stream 35 from the bottom of the scrubber 32 and stream 49 from the suspension-based melt crystallization system 46. The resulting mixture in stream 3 is pre-heated in a pre-heater 4 to a temperature above the melting point of glycolic acid before entering the first reactive distillation column 6. The rising vapors in the distillation column 6 primarily composed of water, are condensed in the condenser 7. The condensate is split into an overhead liquid product stream 9 and a reflux stream 8, which is returned to the top of the column 6. Uncondensed vapors are removed via stream 10 by a vacuum system 11. Glycolic acid and glycolic acid oligomers accumulate at the bottom of the column 6 and flow back to the pot 12. The bottom stream 14 from pot 12 is pumped via the pump 15 and divided into two streams: a bottom product stream 17 and a recirculation stream 16 that is sent to the top of the falling film evaporator 13. In the evaporator 13, the reaction solution partially vaporizes and then returns to the pot 12, where the generated vapors separate from the liquid in the pot 12, with the vapors ascending through the column 6 and the liquid remaining in the pot 12.

A small portion of stream 1 is diverted as split stream 40 to the concentration distillation column 41, where it is refined into a concentrated glycolic acid solution, extracted via stream 42, while water is removed through the overhead stream 43. Any uncondensed vapors are evacuated through stream 44 by the vacuum system 45. The concentrated glycolic acid in stream 42 is then mixed with the bottom product from stream 17 and the depolymerization catalyst from stream 39 in the static mixer 18, forming Stream 19. The bottom stream 21 from pot 20 is transferred via pump 22 and split into a purge stream 23 and a recirculation stream 24. This recirculation stream combines with Stream 19, forming the reaction feed, which is then fed to the top of the falling film evaporator 26. After partial vaporization, the reaction solution proceeds to the pot 20, where vapors separate from the liquid in the pot 20, with the vapors ascending through the column 27 and the liquid remaining in the pot 20. The overhead vapors, which contain a high concentration of glycolide along with some glycolic acid dimers, unreacted glycolic acid, and water, are condensed in the condenser 28. The condensate is divided into an overhead liquid product stream 30 and a reflux stream 29, which returns to the top of the column 27. Uncondensed vapors are removed via stream 31. Unconverted glycolic acid oligomers concentrate at the bottom of the column 27 and flow back to the pot 20.

A concentrated methyl glycolate solution is continuously introduced via stream 36 to a cooler 37 before feeding to the top of the scrubber 32, where it helps remove glycolic acid and glycolic acid dimers from stream 31, which enters the bottom of the scrubber. The resulting liquid stream

35 is recycled back to mix with stream 1 in the static mixer 2. Uncondensed vapors from the top of the scrubber 32 are removed via stream 33 by a vacuum system 34.

The overhead product stream 30 from the top of the distillation column 27 is fed to the suspension-based melt crystallization system 46. The impurity-rich mother liquor stream 47 is divided into a purge stream 48 and a stream 49 which is recycled back to the static mixer 2. Ultrapure glycolide product is extracted via stream 50 for further processing.

FIG.2 illustrates a preferred system for glycolide production system using methyl glycolate solution as the starting material in accordance with the present invention. The system includes a first reactive distillation column 56, a condenser 57, a pot 62, a falling film evaporator 63, a pump 65, a second reactive distillation column 77, a condenser 78, a pot 70, a falling film evaporator 76, a pump 72, a cold trap 82 and a suspension-based melt crystallization system 91.

A concentrated methyl glycolate solution is continuously introduced via stream 51 into a static mixer 52, where it together with a catalyst stream 89 combines with stream 85 from the bottom of the cold trap 82 and stream 94 from the suspension-based melt crystallization system 91. The resulting mixture in stream 53 is pre-heated in a pre-heater 54 before entering the first reactive distillation column 56. The rising vapors in the distillation column 56 primarily composed of methanol, are condensed in the condenser 57. The condensate is split into an overhead liquid product stream 59 and a reflux stream 58, which is returned to the top of the column 56. Uncondensed vapors are removed via stream 60 by a vacuum system 61. Methyl glycolate and methyl glycolate oligomers accumulate at the bottom of the column 56 and flow back to the pot 62. The bottom stream 64 from the pot 62 is pumped via the pump 65 and divided into two streams: a bottom product stream 67 and a recirculation stream 66 that is sent to the top of the falling film evaporator 63. In the evaporator 63, the reaction solution partially vaporizes and then returns to the pot 62, where the generated vapors separate from the liquid in the pot 62, with vapors ascending through the column 56 and liquid remaining in the pot 62.

A small portion of stream 51 is diverted as the split stream 86, which is pre-heated in a pre-heater 87. The heated concentrated methyl glycolate in stream 88 is mixed with the bottom product from stream 67 and the depolymerization catalyst from stream 90 in a static mixer 68, forming stream 69. The bottom stream 71 from the pot 70 is transferred via the pump 72 and split into a purge stream 73 and a recirculation stream 74. This recirculation stream combines with stream 69, forming the reaction feed, which is then fed to the top of the falling film evaporator 76. After partial vaporization, the reaction solution proceeds to the pot 70, where the generated vapors separate from the liquid in the pot 70, with the vapors ascending through the column 77 and the liquid remaining in the pot 70. The overhead vapors, which contain a high concentration of glycolide along with some methyl glycolate dimers, unreacted methyl glycolate, and methanol, are condensed in the condenser 78. The condensate is divided into an overhead liquid product stream 80 and a reflux stream 79, which returns to the top of the column 77. Unconverted methyl glycolate oligomers concentrate at the bottom of the column 77 and flow back to the pot 70. Uncondensed vapors are removed via stream 81 and fed to the cold trap 82, where the vapors are further condensed. The resulting liquid stream 85 is recycled back to mix

with stream 51 in the static mixer 52. Uncondensed vapors from the cold trap 82 are removed via stream 83 by a vacuum system 84.

The overhead product stream 80 from the top of the distillation column 77 is fed to the suspension-based melt crystallization system 91. The impurity-rich mother liquor stream 92 is divided into a purge stream 93 and a stream 94 which is recycled back to the static mixer 52. Ultrapure glycolide product is extracted via stream 95 for further processing.

FIG. 3 illustrates an alternative system for glycolide production using glycolic acid solution as the starting material in accordance with the present invention. Unlike the process shown in FIG. 1, this system introduces a key modification: the split stream 40, which was previously diverted from stream 1 and processed through concentration column 41, is now replaced by a separate stream 101 and a preheater 102. Stream 101 undergoes preheating in the preheater 102, resulting in a heated stream 103, which is then merged with stream 17 and stream 39 to form stream 19. The bottom stream 21 from pot 20 is directed through pump 22, where it is divided into a purge stream 23 and a recirculation stream 24. The recirculation stream 24 merges with stream 19 before being introduced at the top of the falling film evaporator 26. Stream 101 can consist of either a methyl glycolate solution alone or a mixture of glycolic acid and methyl glycolate solution. All other streams remain unchanged from the original process.

Subsequently, the present invention is illustrated in more details below with reference to the drawing and the examples.

EXAMPLES

Example 1

A reactive distillation of the first reactive distillation system according to an embodiment of the invention as shown in FIG.1 was performed. The distillation column 6 had a total of 10 theoretical stages. The aqueous solution of glycolic acid from stream 1, containing 70% glycolic acid by weight and a mass flow rate of 500 kg/h, was combined with streams 35 and 49 within the static mixer 2. The resulting mixture, stream 3, was pre-heated to 100°C via the pre-heater 4 before entering the distillation column 6. During the pre-polymerization step, glycolic acid oligomers were formed and byproduct water was distilled off. The necessary heat for the process was provided by the falling film evaporator 13, which raised the reaction solution temperature up to 210°C. The overhead product stream 9, primarily consisting of water with a mass flow rate of 238 kg/h, was removed for further water treatment. The bottom product stream 17, containing a significant portion of glycolic acid oligomers, was obtained. The distillation column 6 operated with an overhead pressure (pressure of condenser 7) of 40 mbar and a bottom pressure of 43 mbar. Titration of the acid group content in the bottom product showed that the glycolic acid oligomers had an average number molecular weight of 800 g/mol.

A split stream 40 with a mass flow rate of 17 kg/h was fed to the concentration distillation column 41, where it was refined into a glycolic acid solution containing 96% glycolic acid by weight and extracted via stream 42. The distilled water is removed through the overhead stream 43. Any uncondensed vapors are evacuated through stream 44 by the vacuum system 45. The bottom product stream 17 from the first reactive distillation system was combined with the catalyst (tin dioctoate) from stream 39 and the concentrated glycolic acid solution from stream 42 in the static mixer 18, creating stream 19. This resulting stream 19 was then merged with the recirculation stream 24. The combined stream was subsequently fed into the top of the falling film evaporator 26. The glycolide, along with small amounts of formed glycolic acid dimers, water and unreacted glycolic acid was distilled off via stream 30 while the evaporator heated the reaction solution up to 230°C. The overhead product stream 30, with a mass flow rate of 372 kg/h and containing 93.2% glycolide by weight, was collected. The stream 23 was discharged as a purge stream. The distillation column 27 had a total of 7 theoretical stages and operated with an overhead pressure (pressure of condenser 28) of 5 mbar and a bottom pressure of 8 mbar.

The scrubber 32 had a total of 6 theoretical stages. A concentrated methyl glycolate stream containing 97% methyl glycolate by weight was first cooled in the cooler 37 before being fed to the top of the scrubber 32, where it utilized an internal pumparound configuration within its stripping section. Unreacted glycolic acid and glycolic acid dimers from stream 31 were absorbed in the cooled methyl glycolate as they entered the bottom of the scrubber. The resulting liquid, with a mass flow rate of 45 kg/h, was then recycled back to mix with stream 1 in the static mixer 2. Any uncondensed vapors from the top of the scrubber were removed via stream 33 by a vacuum system 34.

The overhead product stream 30, which was enriched in glycolide, underwent additional processing in a two-stage suspension-based melt crystallization system 46. This process produced an ultrapure glycolide product with a purity of 99.92% by weight and a mass flow rate of 280 kg/h, which was collected through stream 50.

Example 2

A reactive distillation of the first reactive distillation system according to an embodiment of the invention as shown in FIG.1 was performed. The distillation column 6 had a total of 10 theoretical stages. The aqueous solution of glycolic acid from stream 1, containing 70% glycolic acid by weight and a mass flow rate of 500 kg/h, was combined with streams 35 and 49 within the static mixer 2. The resulting mixture, stream 3, was pre-heated to 100°C via the pre-heater 4 before entering the distillation column 6. During the pre-polymerization step, glycolic acid oligomers were formed and byproduct water was distilled off. The necessary heat for the process was provided by the falling film evaporator 13, which raised the reaction solution temperature up to 215°C. The overhead product stream 9, primarily consisting of water with a mass flow rate of 242 kg/h, was removed for further water treatment. The bottom product stream 17, containing a significant portion of glycolic acid oligomers, was obtained. The distillation column 6 operated with an overhead pressure (pressure of condenser 7) of 40 mbar and a bottom pressure of 43

mbar. Titration of the acid group content in the bottom product showed that the glycolic acid oligomers had an average number molecular weight of 2,000 g/mol.

A split stream 40 with a mass flow rate of 34 kg/h was fed to the concentration distillation column 41, where it was refined into a glycolic acid solution containing 96% glycolic acid by weight and extracted via stream 42. The distilled water is removed through the overhead stream 43. Any uncondensed vapors are evacuated through stream 44 by the vacuum system 45. The bottom product stream 17 from the first reactive distillation system was combined with the catalyst (tin dioctoate) from stream 39 and the concentrated glycolic acid solution from stream 42 in the static mixer 18, creating stream 19. This resulting stream 19 was then merged with the recirculation stream 24. The combined stream was subsequently fed into the top of the falling film evaporator 26. The glycolide, along with small amounts of formed glycolic acid dimers, water and unreacted glycolic acid was distilled off via stream 30 while the evaporator heated the reaction solution up to 240°C. The overhead product stream 30, with a mass flow rate of 386 kg/h and containing 90.9% glycolide by weight, was collected. The stream 23 was discharged as a purge stream. The distillation column 27 had a total of 7 theoretical stages and operated with an overhead pressure (pressure of condenser 28) of 10 mbar and a bottom pressure of 13 mbar.

The scrubber 32 had a total of 6 theoretical stages. A concentrated methyl glycolate stream containing 97% methyl glycolate by weight was first cooled in the cooler 37 before being fed to the top of the scrubber 32, where it utilized an internal pumparound configuration within its stripping section. Unreacted glycolic acid and glycolic acid dimers from stream 31 were absorbed in the cooled methyl glycolate as they entered the bottom of the scrubber. The resulting liquid, with a mass flow rate of 55 kg/h, was then recycled back to mix with stream 1 in the static mixer 2. Any uncondensed vapors from the top of the scrubber were removed via stream 33 by a vacuum system 34.

The overhead product stream 30, which was enriched in glycolide, underwent additional processing in a two-stage suspension-based melt crystallization system 46. This process produced an ultrapure glycolide product with a purity of 99.90% by weight and a mass flow rate of 284 kg/h, which was collected through stream 50.

Example 3

A reactive distillation of the first reactive distillation system according to an embodiment of the invention as shown in FIG.2 was performed. The distillation column 56 had a total of 10 theoretical stages. The methyl glycolate solution from stream 51, with a concentration of 97% by weight and a mass flow rate of 500 kg/h, was combined with streams 85, 94, and catalyst stream 89 within static mixer 52. The resulting mixture, stream 53, was pre-heated to 70°C via the pre-heater 54 before entering the distillation column 56. During the pre-polymerization step, methyl glycolate oligomers were formed and byproduct methanol was distilled off. The necessary heat for the process was provided by the falling film evaporator 63, which raised the reaction solution temperature up to 210°C. The overhead product stream 59, primarily consisting of methanol with a mass flow rate of 190 kg/h, was removed for further treatment. The bottom product stream 67,

containing a significant portion of methyl glycolate oligomers, was obtained. The distillation column 56 operated with an overhead pressure (pressure of condenser 57) of 200 mbar and a bottom pressure of 203 mbar. Titration of the acid group content in the bottom product showed that the methyl glycolate oligomers had an average number molecular weight of 800 g/mol.

A split stream 86, with a mass flow rate of 12 kg/h and a concentration of 97% methyl glycolate by weight, was pre-heated in a pre-heater 87. The heated concentrated methyl glycolate in stream 88 was mixed with the bottom product from stream 67 and the depolymerization catalyst from stream 90 in a static mixer 68, forming stream 69. The bottom stream 71 from the pot 70 was split into a purge stream 73 and a recirculation stream 74. This recirculation stream combined with stream 69, forming the reaction feed, which was then fed to the top of the falling film evaporator 76. The glycolide, along with small amounts of formed methyl glycolate dimers, methanol and unreacted methyl glycolate was distilled off via stream 80 while the evaporator heated the reaction solution up to 230°C. The overhead product stream 80, with a mass flow rate of 388 kg/h and containing 94.9% glycolide by weight, was collected. The distillation column 77 had a total of 7 theoretical stages, which operated with an overhead pressure (pressure of condenser 78) of 8 mbar and a bottom pressure of 11 mbar.

Uncondensed vapors were removed via stream 81 and fed to the cold trap 82, where the vapors were further condensed. The resulting liquid stream 85 with a mass flow rate of 8 kg/h was recycled back to mix with stream 51 in the static mixer 52. Uncondensed vapors from the cold trap 82 were removed via stream 83 by the vacuum system 84.

The overhead product stream 80, which was enriched in glycolide, underwent additional processing in a two-stage suspension-based melt crystallization system 91. This process produced an ultrapure glycolide product with a purity of 99.93% by weight and a mass flow rate of 298 kg/h, which was collected through stream 95.

Example 4

A reactive distillation of the first reactive distillation system according to an embodiment of the invention as shown in FIG.2 was performed. The distillation column 56 had a total of 10 theoretical stages. The methyl glycolate solution from stream 51, with a concentration of 97% by weight and a mass flow rate of 500 kg/h, was combined with streams 85, 94, and catalyst stream 89 within static mixer 52. The resulting mixture, stream 53, was pre-heated to 70°C via the pre-heater 54 before entering the distillation column 56. During the pre-polymerization step, methyl glycolate oligomers were formed and byproduct methanol was distilled off. The necessary heat for the process was provided by the falling film evaporator 63, which raised the reaction solution temperature up to 215°C. The overhead product stream 59, primarily consisting of methanol with a mass flow rate of 194 kg/h, was removed for further treatment. The bottom product stream 67, containing a significant portion of methyl glycolate oligomers, was obtained. The distillation column 56 operated with an overhead pressure (pressure of condenser 57) of 200 mbar and a bottom pressure of 203 mbar. Titration of the acid group content in the bottom product showed that the methyl glycolate oligomers had an average number molecular weight of 2,000 g/mol.

A split stream 86, with a mass flow rate of 24 kg/h and a concentration of 97% methyl glycolate by weight, was pre-heated in a pre-heater 87. The heated concentrated methyl glycolate in stream 88 was mixed with the bottom product from stream 67 and the depolymerization catalyst from stream 90 in a static mixer 68, forming stream 69. The bottom stream 71 from the pot 70 was split into a purge stream 73 and a recirculation stream 74. This recirculation stream combined with stream 69, forming the reaction feed, which was then fed to the top of the falling film evaporator 76. The glycolide, along with small amounts of formed methyl glycolate dimers, methanol and unreacted methyl glycolate was distilled off via stream 80 while the evaporator heated the reaction solution up to 240°C. The overhead product stream 80, with a mass flow rate of 396 kg/h and containing 92.4% glycolide by weight, was collected. The distillation column 77 operated with an overhead pressure (pressure of condenser 78) of 15 mbar and a bottom pressure of 18 mbar.

Uncondensed vapors were removed via stream 81 and fed to the cold trap 82, where the vapors were further condensed. The resulting liquid stream 85 with a mass flow rate of 10 kg/h was recycled back to mix with stream 51 in the static mixer 52. Uncondensed vapors from the cold trap 82 were removed via stream 83 by the vacuum system 84.

The overhead product stream 80, which was enriched in glycolide, underwent additional processing in a two-stage suspension-based melt crystallization system 91. This process produced an ultrapure glycolide product with a purity of 99.91% by weight and a mass flow rate of 296 kg/h, which was collected through stream 95.

Example 5

A reactive distillation of the first reactive distillation system according to an embodiment of the invention as shown in FIG.3 was performed. The distillation column 6 had a total of 10 theoretical stages. The aqueous solution of glycolic acid from stream 1, containing 70% glycolic acid by weight and a mass flow rate of 500 kg/h, was combined with streams 35 and 49 within the static mixer 2. The resulting mixture, stream 3, was pre-heated to 100°C via the pre-heater 4 before entering the distillation column 6. During the pre-polymerization step, glycolic acid oligomers were formed and byproduct water was distilled off. The necessary heat for the process was provided by the falling film evaporator 13, which raised the reaction solution temperature up to 212°C. The overhead product stream 9, primarily consisting of water with a mass flow rate of 240 kg/h, was removed for further water treatment. The bottom product stream 17, containing a significant portion of glycolic acid oligomers, was obtained. The distillation column 6 operated with an overhead pressure (pressure of condenser 7) of 40 mbar and a bottom pressure of 43 mbar. Titration of the acid group content in the bottom product showed that the glycolic acid oligomers had an average number molecular weight of 1,000 g/mol.

A stream 101, with a mass flow rate of 20 kg/h and a concentration of 97% methyl glycolate by weight, was pre-heated in a pre-heater 102. The heated concentrated methyl glycolate in stream 103 was mixed with the bottom product from stream 17 and the depolymerization catalyst from stream 39 in a static mixer 18, forming stream 19. The bottom stream 21 from the pot 20 was

split into a purge stream 23 and a recirculation stream 24. This recirculation stream combined with stream 19, forming the reaction feed, which was then fed to the top of the falling film evaporator 26. The glycolide, along with small amounts of formed methyl glycolate dimers, water and unreacted glycolic acid was distilled off via stream 30 while the evaporator heated the reaction solution up to 230°C. The overhead product stream 30, with a mass flow rate of 392 kg/h and containing 91.3% glycolide by weight, was collected. The distillation column 27 had a total of 7 theoretical stages and operated with an overhead pressure (pressure of condenser 28) of 7 mbar and a bottom pressure of 10 mbar.

The scrubber 32 had a total of 6 theoretical stages. A concentrated methyl glycolate stream containing 97% methyl glycolate by weight was first cooled in the cooler 37 before being fed to the top of the scrubber 32, where it utilized an internal pumparound configuration within its stripping section. Unreacted glycolic acid, dimers and methyl glycolate from stream 31 were absorbed in the cooled methyl glycolate as they entered the bottom of the scrubber. The resulting liquid, with a mass flow rate of 60 kg/h, was then recycled back to mix with stream 1 in the static mixer 2. Any uncondensed vapors from the top of the scrubber were removed via stream 33 by a vacuum system 34.

The overhead product stream 30, which was enriched in glycolide, underwent additional processing in a two-stage suspension-based melt crystallization system 46. This process produced an ultrapure glycolide product with a purity of 99.91% by weight and a mass flow rate of 288 kg/h, which was collected through stream 50.

Example 6

A reactive distillation process was conducted using the first reactive distillation system as illustrated in FIG. 3, according to an embodiment of the invention. A glycolic acid oligomer with an average number molecular weight of 1,000 g/mol was produced, following the same procedure as detailed in Example 5. The depolymerization and purification steps were carried out under identical conditions to those described in example 5, with the exception that stream 101 consisted of a mixture of concentrated glycolic acid (96% by weight) and methyl glycolate solution (97% by weight), at a total mass flow rate of 30 kg/h. In this mixture, the concentrated glycolic acid solution comprised 20% of the total mass, while the methyl glycolate solution accounted for the remaining 80%.

The overhead product stream 30, which was enriched in glycolide, underwent additional processing in a two-stage suspension-based melt crystallization system 46. This process produced an ultrapure glycolide product with a purity of 99.90% by weight and a mass flow rate of 294 kg/h, which was collected through stream 50.

The invention claimed is:

1. A method for the continuous production of glycolide, comprising: a pre-polymerization step wherein glycolic acid or methyl glycolate is pre-polymerized into their respective oligomers; a depolymerization step wherein a reactive distillation system is utilized to depolymerize said oligomers into crude glycolide, characterized in that a small quantity of liquid substance is introduced, as part of the reaction feed, into the depolymerization reactor; and a purification step wherein said crude glycolide is processed to ultrapure glycolide through a melt crystallization system.
2. The method of claim 1, wherein said oligomers have an average number molecular weight ranging from 400 to 5,000, preferably ranging from 600 to 3,000, and more preferably ranging from 800 to 2,000.
3. The method of claim 1, wherein said reactive distillation system comprises at least a pot, a distillation column, a horizontal condenser, and a falling film evaporator.
4. The method of claim 3, wherein said distillation column and falling film evaporator are preferably mounted on top of said pot, respectively, with said horizontal condenser installed on top of said distillation column.
5. The method of claim 1, wherein said liquid substance is selected from the group consisting of concentrated glycolic acid solution, concentrated methyl glycolate solution, or a combination thereof.
6. The method of claim 1, wherein said liquid substance content in said oligomers produced during said pre-polymerization step is preferably between 1% and 10% by weight, with a more preferred range of 3% to 6% by weight.
7. The method of claim 1 and 3, wherein said depolymerization reactor is said falling film evaporator.
8. The method of claim 3, wherein a bottom stream from said pot is split into a purge stream and a recirculation stream.
9. The method of claims 1 and 8, wherein said reaction feed comprises a mixture of said liquid substance, a depolymerization catalyst, said oligomers from said pre-polymerization step, and said recirculation stream.
10. The method of claim 1, wherein said reaction feed is introduced at the top of said depolymerization reactor, flowing downward through long vertical tubes that function as the heat transfer and reaction zone, with these tubes being surrounded by a heat transfer medium.
11. The method of claim 1, wherein said crude glycolide consists primarily of glycolide produced in the depolymerization reactor, along with a smaller portion of low-boiling point volatiles, including said liquid substance, its dimers, and associated byproducts such as water and methanol.

12. The method of claim 1, wherein said melt crystallization system is preferably a suspension-based melt crystallization system.
13. The method of claim 12, wherein said suspension-based melt crystallization system comprises at least one wash column for producing ultrapure glycolide.
14. The method of claim 12, wherein the mother liquor from said suspension-based melt crystallization system is recycled back into said pre-polymerization step.
15. The method of claim 1 and 3, wherein the operating pressure of said horizontal condenser in said reactive distillation system is set within a range of 3 to 30 mbar.