INVITED REVIEW

Development of an industrial production technology for high-molecular-weight polyglycolic acid

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Polyglycolic acid (PGA), a biodegradable aliphatic polyester, has only been produced in small quantities as an extremely expensive, high value-added product because no technology has existed to permit inexpensive mass production. PGA is a novel biodegradable resin that offers high mechanical strength and high gas-barrier performance. To mass-produce high-molecular-weight PGA on an industrial scale, Kureha Corporation has developed a process to obtain large yields of the intermediate glycolide (GL) with high levels of purity. Using the obtained GL, we developed a method to polymerize high-molecular-weight PGA continuously. A commercial production plant is now in operation. Because high-molecular-weight PGA can be produced at a lower cost than previously possible, we have also developed various applications that utilize its characteristics. The use of PGA in shale gas and oil exploration is of interest because PGA can supply ultra-strong and biodegradable materials.

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INTRODUCTION

Polyglycolic acid (PGA) has the simplest chemical structure of the aliphatic polyesters, as shown in Figure 1, and was first synthesized in 1932 by Carothers.

However, high-molecular-weight PGA could not be obtained because it was unstable and easily degradable compared with other synthetic polymers.¹ In the 1950s, DuPont discovered a method for synthesizing high-molecular-weight PGA through the ring-opening polymerization of glycolide (GL), the cyclic di-ester of glycolic acid (Figure 2).²

In the 1960s, an American company focused on the biodegradability and the biocompatibility of PGA and successfully used PGA as a bioabsorbable suture thread.³ PGA and its copolymers with polylactic acid have been used subsequently as components in regenerative medical materials. However, without a low-cost technology for mass-producing PGA, only a small volume can be produced at a high cost. Consequently, the application of PGA as a high value-added product has been limited to the field of medicine.

We at the Kureha Corporation (KUREHA) have been conducting fundamental research on PGA as a biodegradable polymer since the mid-1990s.⁴ At that time, we were manufacturing various synthetic polymers while societal concern regarding environmental consciousness was growing. We were confident that a market for environmentally friendly, biodegradable polymers would eventually emerge. In addition to the biodegradability of PGA, we quickly realized that its peculiar molecular structure possesses some notable properties, such as high gas-barrier characteristics and mechanical strength, surpassing that of super-engineering plastics. We anticipated that if we could overcome the conventional difficulties in PGA manufacturing and supply high-molecular-weight PGA at a

reasonable price, then PGA could be used as a novel multifunctional biodegradable polymer.

In this review, we discuss the industrial production technology that KUREHA has established for producing high-molecular-weight PGA. We also discuss new applications that we have developed for PGA as a highly functional, environmentally friendly, biodegradable polymer.

INDUSTRIAL MANUFACTURING METHOD FOR GL

The simplest method for synthesizing PGA is through the condensation of glycolic acid using a dehydrating reaction. However, because there is an equilibrium between GL generation and the chain extension for the hydroxyl termination of PGA, sufficiently high molecular weights cannot be achieved using this method.

Eventually, a method of synthesizing high-molecular-weight PGA through the ring-opening polymerization of GL was discovered, and alternative methods were unknown until now. Therefore, high-purity GL had been essential for obtaining high-molecular-weight PGA.

The conventional manufacturing method for GL entails melting a glycolic acid oligomer (GAO) by heating and then recovering, through evaporation, the GL as a depolymerization product from the surface of the melted GAO. However, this method could not be used for mass production for the following reasons:

(1) The melted GAO is unstable and highly viscous. Because heat transfer efficiency is difficult to improve, wall surface temperature must be increased. Consequently, adverse reactions, such as tar formation, can occur easily.

(2) Evaporated GL easily deposits on the inside walls of distillation lines. Because this incrustation promptly polymerizes, distillation lines easily become obstructed.

Figure 1 Polyglycolic acid. **Figure 2** Production flow of polyglycolic acid (PGA). GL, glycolide. A full color version of this figure is available at *Polymer Journal* online. **Figure 3** Graphical schema of depolymerization. GL, glycolide.

KUREHA overcame these issues by implementing an original method and process.^{5,6} Specifically, GAO is melted through heating in a novel solvent developed for this technology. This melting forms a uniform, low-viscosity and liquid solution. Through distillation, the GL generated by the depolymerization reaction is recovered along with the special solvent (Figure 3).

The GAO depolymerization reaction to generate GL is an intramolecular cyclization reaction because of the backbiting of the hydroxyl-terminated group in GAO, and there is a ring-chain equilibrium between GL and GAO (Figure 4).

Because of the equilibrium, a dilute solution is more favorable in terms of reaction efficiency. By transferring GL to the gas phase with the solvent, the GL generation efficiency increases, and its residence time in the vessel decreases. Consequently, adverse heavy reactions are suppressed. Distillation line obstruction is also suppressed because GL is vaporized with the solvent. These advantages form the core factors of this technology and are particularly beneficial when the process is scaled up (Table 1).

The solvent used in this process is crucial. It must be capable of dissolving GAO at high temperatures; must have a suitable vapor pressure for GL evaporation; must remain stable under

Figure 4 Ring-chain equilibrium between glycolide (GL) and glycolic acid oligomer (GAO).**Table 1** Comparison of the depolymerization processes between current and Kureha

	Current	Kureha
Phase	Bulk	In solvent
Temperature	> 280 °C	210–260 °C
Pressure	< 0.1 kPa	1–10 kPa
Viscosity	High	Low
Remarks	Low productivity Frequent clean-up	Easy scale-up Easy long run

high-temperature reaction conditions for long periods; must form an azeotrope with GL; and must have an appropriate solubility after condensation to permit the easy separation of GL. Thus, a solvent with properties consistent with these requirements, such as poly alkylene glycol di-ether, was designed and synthesized in-house to develop the correct solution-method depolymerization process.

To facilitate inexpensive industrial mass production, we also addressed impurities in the raw material. The raw material, glycolic acid, contains diglycolic acid (HOOCCH₂OCH₂COOH) that seals the hydroxy termini of GAO, thereby suppressing the depolymerization reaction. This suppression was overcome by adding alcohol⁷ that not only restored the OH end group as the starting point of depolymerization but also improved the solubility of GAO in the solvent.

INDUSTRIAL MANUFACTURING METHOD FOR HIGH-MOLECULAR-WEIGHT PGA

High-molecular-weight PGA is generally synthesized through a ring-opening polymerization from highly purified GL in the bulk condition. In the conventional polymerization method, the melt viscosity of the generated polymer increases significantly as the polymerization reaction progresses, thus creating difficulties in terms of agitation in the reactor and discharge from the reactor. Therefore, high-molecular-weight PGA has been manufactured in batches using small-scale apparatuses.

To overcome these difficulties, we developed a new method for synthesizing high-molecular-weight PGA on an industrial scale.^{8,9} In this method, the ring-opening polymerization of GL is achieved within the temperature range between the melting point of GL (~90 °C) and the melting point of high-molecular-weight PGA (~220 °C). This polymerization reaction is initially induced in the molten state, and after reaching an appropriate reaction conversion, the polymer is precipitated as a solid. Then, by promoting polymerization in the solid state, high-molecular-weight PGA is generated. This method enables the production of a polymer with a size and shape suitable for subsequent manufacturing processes (Table 2).

We performed studies to identify the factors that control the reaction rate, the molecular weight of the generated polymer and the terminating groups' structures. Based on these factors,^{10,11} we established a technology that could be used on an industrial scale by controlling the polymerization reaction during the initial reaction period, by controlling the phase transition from the molten

Table 2 Comparison of the polymerization processes between classic, current and Kureha

	Ring-opening polymerization of glycolide		Dehydration–condensation of glycolic acid
	Current	Kureha	Classic
Temperature (°C)	≥220	150–200	≥220
Phase	Molten	Molten to solid	Molten
Handling of the polymer	Difficult to remove from reactor for high viscosity	Easy to remove as a pulverized solid	Difficult to remove from reactor for high viscosity
Physical properties	Colored (brown) Middle molecular weight (Mw; ≤20E4)	White color High molecular weight (Mw; ≤50E4)	Colored (brown) Small molecular weight (Mw; 2–3E4)

Figure 5 Range of molding temperatures for polyglycolic acid (PGA) and other polymers. A full color version of this figure is available at *Polymer Journal* online.

state to the solid state during the intermediate period and by cooperating with the polymerization conditions in the solid state.

IMPROVING HIGH-MOLECULAR-WEIGHT PGA

Conventional PGA is usually unsatisfactory in terms of thermal stability; therefore, its heat resistance must be improved to prepare PGA for applications in several fields in conjunction with melt-molding technology. An examination of the thermal decomposition mechanism of PGA identified compounds with a heat stabilizer, such as the deactivator of the residual catalyst that effectively improves its heat resistance. Through the reactive processing of these compounds with PGA in an extruder, we improved the heat resistance of PGA without altering its basic properties, thus making PGA viable for melt-molding processes in various applications (Figure 5).¹²

We also developed technology to control the hydrolyzability of PGA for applications that require the long-term retention of PGA properties. We determined that the hydrolysis rate of PGA could be decreased primarily by controlling the structure of the termini of the PGA polymer and by reducing the small amounts of residual GL in the polymer.¹³ We were able to decrease and to repress the hydrolysis rate by an order of magnitude, thereby making PGA commercially viable for use in applications that require the long-term retention of PGA properties. The structures of PGA polymer termini can be controlled effectively in two ways: (1) regulating the types of alcohols used as initiators in polymerization and controlling the concentrations at which they are used, and (2) using polymer reactions¹⁴ for compounds that react with the terminal species of the generated polymer. Both of these controls were introduced into

Figure 6 OTR and WVTR of polyglycolic acid (PGA) and other polymers. OTR, oxygen transmission rate; WVTR, water vapor transmission rate. A full color version of this figure is available at *Polymer Journal* online.

the polymer manufacturing process. These controls do not affect properties such as the degree of crystallinity and the melting point.

Residual GL can be removed from the polymer under normal heating conditions, but if this operation is performed on the polymer immediately after polymerization is complete, depolymerization occurs, and the final molecular weight is reduced. The compounds introduced to improve heat resistance, as described above, also serve to suppress depolymerization during this operation, thereby reducing the amount of residual GL.

CHARACTERISTICS AND NEW APPLICATIONS OF HIGH-MOLECULAR-WEIGHT PGA

Applications for conventional high-molecular-weight PGA were restricted to the medical field because of the extremely high price of PGA. The relevant characteristics were its biodegradability and biocompatibility. By focusing on the molecular structure of PGA, we discovered that it possesses other noteworthy properties. In particular, its gas-barrier characteristics and mechanical strength are the highest among existing polymers. These properties led us to believe that new applications could be developed in which PGA is a highly functional and unique biodegradable polymer. We describe its further evolution below:

Gas-barrier characteristics

Because of its molecular structure, PGA has a small free volume and high gas-barrier properties (100 times that of polyethylene terephthalate (PET); Figure 6).

Figure 7 Effect of polyglycolic acid (PGA) density on oxygen transmission rate (OTR). A full color version of this figure is available at *Polymer Journal* online.

Figure 8 Multilayer polyethylene terephthalate (PET) bottles manufactured with polyglycolic acid (PGA).

The high density of PGA leads to high gas-barrier performance (Figure 7). The density of PGA depends on the crystallinity of PGA.

One application that takes advantage of these properties is the production of PET bottles used for storing and transporting carbonated beverages.^{15,16} In a multilayer PET/PGA/PET bottle (Figure 8), the rate of carbonic acid loss from the beverage is significantly decreased compared with that in traditional single-layer PET bottles.

PGA in the multilayer PET/PGA/PET bottle must have mechanical strength to function properly as a gas barrier. PGA has been able to maintain a high molecular weight and mechanical strength during the shelf life of the beverage.

Furthermore, by inserting PGA as an intermediate layer, it is possible to decrease the weight of the bottle while extending the shelf life of the beverage. A recycling system has already been established for PET bottles in which the bottles are pulverized into flakes and then cleaned, and the PET is dried and subsequently reused as recycled PET. In this process, the PGA layer in multilayer PET/PGA/PET bottles could be separated easily from the PET layer; it has been demonstrated that the recycled PET does not contain any PGA.^{17,18}

Other applications that utilize the gas-barrier characteristics of PGA include multilayer/blend films and cups.^{19–21} It is possible to form a high gas barrier using a composite with paper²² that is highly promising as a green plastic material.

High mechanical strength

Because PGA is crystalline and has a high density, its mechanical strength equals or exceeds that of other existing resins. As a high-performance resin (engineering plastic), the strength of PGA is approximately equivalent to that of the polyether ether ketone resin used for automotive parts (Figure 9).

PGA is essentially hydrolyzable and is ranked highly as a distinctive high-performance material. Nonetheless, its durability in

Figure 9 Flexural strength. A full color version of this figure is available at *Polymer Journal* online.

Figure 10 Schematic cross-section of the subsurface illustrating types of natural gas deposits. A full color version of this figure is available at *Polymer Journal* online.

Figure 11 Polyglycolic acid (PGA) plant in West Virginia, USA.

high-temperature and high-humidity environments, which promote hydrolysis, must be considered.

Degradability

PGA has attracted considerable attention as an auxiliary material for use in shale gas/oil well drilling or completion work. Shale gas/oil is produced from lateral wellbores that reach deep underground (Figure 10).²³

Although conventional gas/oil is easily recovered from the reservoir because of its high permeability, a shale gas/oil reservoir with low permeability requires a stimulation job such as fracturing that can insert fractures into the reservoir by injecting a highly viscous

Figure 12 Polyglycolic acid (PGA) is recognized as a biodegradable polymer. A full color version of this figure is available at *Polymer Journal* online.

water-based fluid. Approximately 20 stages of fracturing per well are conducted, and these stages require zonal isolation (that is, plugging the previous fracture zone to prevent a loss of pressure for diverting the fluid to create another targeted fracture). To facilitate this prevention and to prevent fluid loss during drilling, the temporary plugging/fluid loss agent is utilized to enhance work efficiency. This 'temporary' agent must disappear within a certain time period after performing its function to prevent damage to the hole and must be less harsh to the environment. Along with its environmental safety, the hydrolyzability of PGA could be effectively used to satisfy the requirements even at relatively low temperatures. PGA can also function as a delayed-release acid through its degradation, thus promoting the decomposition of ingredients in the fluid (for example, by lowering the viscosity of the fluid). Therefore, PGA could help to enhance productivity by directly improving economic efficiency in the recovery of shale gas/oil.

Given its competitive edge with respect to degradability and its mechanical strength, PGA is also promising as a raw material for creating specialty parts used in downhole tools. Therefore, the process of breaking or recovering PGA tools remaining in the downhole can be eliminated. Moreover, PGA tools cannot be production barriers because they do not leave debris in wellbore. This application is expected to demonstrate the evolution of PGA as an industrial product based on its useful characteristics as a degradable polymer material. We also believe that PGA could contribute significantly to solving the energy problems that we face today.^{24–26}

Others

The properties of PGA make it useful for several other applications beyond those mentioned above. Examples include industrial materials or auxiliary materials, such as masking or etching agents,^{27,28} as well as films and sheets that require high-strength materials.

INDUSTRIALIZATION AND COMMERCIALIZATION OF HIGH-MOLECULAR-WEIGHT PGA

In 2002, at the KUREHA Iwaki facility in Fukushima, we started a pilot plant operation to produce a yearly output of 100 tons and progressed with the development of applications. In 2011, at the DuPont Belle plant in West Virginia, USA, a commercial plant began operation with an annual production of 4000 tons (Figure 11).^{29,30}

Sales of the product began in 2010, and the product was applied in the manufacturing of other products such as bottles, oil drilling tools and suture threads. Various new applications are under development.

The total number of domestic and foreign patents based on this technology exceeds 770, and the total number of patent rights exceeds 230. These numbers far exceed those of other companies in Japan and abroad. The number of patent applications for inventions aimed at improving the manufacturing process and inventions with applications in various fields is increasing daily. A technological infrastructure has been established to prevent other companies from duplicating our novel methods.

CONCLUSION

KUREHA has established an industrial manufacturing technology for high-molecular-weight PGA and has begun commercial production of this material. Based on the characteristics of PGA, we have also developed new applications for PGA as a multifunctional biodegradable polymer (Figure 12).

PGA is universally recognized as an environmentally friendly, highly functional resin, and we expect that it will contribute significantly to society across a broad range of fields.

ACKNOWLEDGEMENTS

In recognition of our remarkable progress and success in the development of polymer technologies and for the launch of a new PGA business, we have received an award from the Society of Polymer Science of Japan (2011). The work described in the article 'Development of Mass Production Technology of High-Molecular-Weight Polyglycolic Acid and Its Application as Advanced Biodegradable Material' was also recognized as an outstanding technology. KUREHA has also received two other awards: the SCEJ Technology Award from The Society of Chemical Engineers of Japan (2011) and the 2011 Best New Product Award Finalist from EDISON AWARDS. Recently, Kureha received the Minister of Economy, Trade and Industry's Prize in the Product and Technology Development Category of the 5th Monodzukuri Nippon Grand Award for developing an environmental load-reducing, high-performance biodegradable polymer, polyglycolic acid (PGA). These awards are because of the ceaseless efforts and contributions of many individuals in the Kureha Group (KUREHA Corp., Kureha Engineering Co. Ltd, Kureha Special Laboratory Co. Ltd, Kureha Gosen Co. Ltd and Kureha Extron Co. Ltd, KUREHA PGA LLC and KUREHA AMERICA LLC in the United States and KUREHA EUROPE B.V. in The Netherlands). We express our deepest gratitude to all members who have been passionate about the development and commercialization of our new PGA and who have contributed to this success.

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