

BioPBS™ (Polybutylene Succinate)



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Abstract PBS (polybutylene succinate) is a biodegradable polymer that is spontaneously degraded into water and carbon dioxide due to the power of microorganisms under soil in the natural world. PBS has high heat resistance and good mechanical properties among general biodegradable polymers, is moldable into the molded

articles such as films, sheets, and fibers, and has a characteristic feature such that compatibility with other biodegradable polymers is high.

Mitsubishi Chemical Corporation (MCC) has successfully achieved commercialization of PBS without using a chain extender in a trademark name of “GS Pla™” and commenced the market development since 2003. “GS Pla™” is completely a biodegradable polymer made of fossil-based succinic acid and 1,4-butanediol as the main raw materials.

Since 2017, PTTMCC Biochem, a joint venture established by Mitsubishi Chemical and PTT Global Chemical Public Company Limited in Thailand, has performed continuous commercial production of bio-based PBS (trademark: BioPBS™) made of bio-based succinic acid and fossil-based 1,4-butanediol.

Mitsubishi Chemical has also been developing, manufacturing, and selling FORZEAS™ that is a compounding material giving new functions to BioPBS™ utilizing the excellent compatibility with various biodegradable materials and biodegradability of BioPBS™. In this article, basic physical properties, biodegradability, moldability, certification acquisition, and characteristic features of BioPBS™ and FORZEAS™ were reviewed.

Keywords 1,4-Butanediol · Bio-based · Biodegradable polymer · Succinic acid

Abbreviations

| | |
|-------|--|
| LLDPE | Linear low density polyethylene |
| PBAT | Poly(butylene adipate-co-butylene terephthalate) |
| PBS | Polybutylene succinate |
| PBSA | Poly(butylene succinate-co-butylene adipate) |
| PBT | Poly butylene terephthalate |
| PCL | Poly(caprolactone) |
| PE | Polyethylene |
| PES | Polyethylene succinate |
| PET | Polyethylene terephthalate |
| PHB | Poly(3-hydroxybutylate) |
| PHB/V | Poly(3-hydroxybutylate-co-3-hydroxyvalerate) |
| PLA | Poly(lactic acid) |
| PP | Polypropylene |
| PS | Polystyrene |
| PVA | Polyvinyl alcohol |
| PVC | Polyvinyl chloride |

1 Introduction

1.1 History of Biodegradable Polymer

While natural polymers such as cellulose, proteins, polysaccharide and natural rubber have been utilized from of old by humankind, synthetic polymers were proposed by H. Staudinger about hundred years ago in 1920. Since then, various polymer materials have been invented and put into practical use in the twentieth century, and almost all of them were non-degradable polymers made of raw materials derived from petroleum (Fig. 1).

In 1932, DuPont commercialized synthetic rubbers and in 1938, nylon. Regarding the aromatic polyesters, ICI commercialized PET in 1949, and Celanese industrialized PBT in 1971. And DuPont commercialized polyacetal in 1956.

Research regarding the aliphatic polyester polymers has a long history. Before the development of PET and PBT, Carothers of DuPont who is famous as the inventor of nylon had performed the research of aliphatic polyesters and reported polycondensation of the aliphatic polyester in 1929 [1]. Later, P.J. Floly successfully increased the molecular weight using an acid chloride as the raw material [2]. However, the aliphatic polyesters were not put into commercial use as the material over a long time because of low heat stability in comparison with aromatic polyesters such as PET and PBT as well as polyamides such as nylon.

In points of mechanical properties, the aliphatic polyesters have lower strength than the aromatic polyesters, it was needed to increase a degree of polymerization in order to put them into practical use. Meanwhile, the low heat stability of the aliphatic

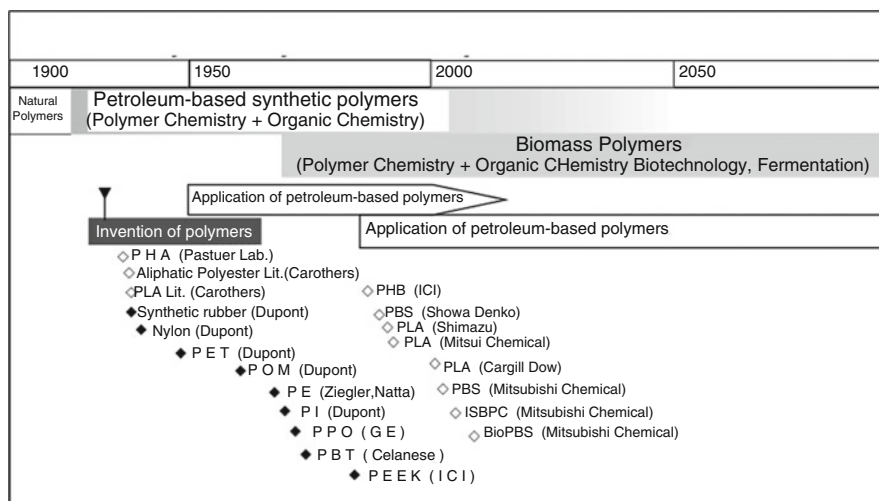


Fig. 1 History of Polymer Industry

polyesters made it difficult to increase the molecular weight by means of melt polycondensation reaction at high temperatures.

For these reasons, it was forced to adopt a manufacturing method by obtaining a polyester having a relatively low molecular weight to some extent, and then connecting the terminals with each other using a chain extender to increase the molecular weight.

1.2 Why PBS among Aliphatic Polyesters?

Because the aliphatic polyesters have biodegradability and many of them are crystalline, it has been proposed that they will possibly have a potential capable of being processed into the molded articles such as films, sheets, and fibers, if it could be achieved to increase the molecular weight. There are three methods for the synthesis of aliphatic polyester: the polycondensation of an aliphatic dicarboxylic acid and an aliphatic diol, the homopolycondensation of an aliphatic oxycarboxylic acid, and the ring-opening polymerization of an aliphatic cyclic ester.

The melting points of various polyesters made of an aliphatic linear dicarboxylic acid and an aliphatic linear diol were summarized in Table 1 [3]. As compared to the melting points of aromatic polyesters, those of aliphatic polyesters are low, and only four types of polyesters having a melting point of higher than 100°C could be made by a combination of oxalic acid or succinic acid with ethylene glycol or 1,4-butanediol. Because the polycondensation using oxalic acid as the raw material is technically difficult for industrial production, polybutylene succinate made of succinic acid and 1,4-butanediol as raw materials, which has highest heat resistance and is advantageous for industrial production, was considered to be the most promising biodegradable polymer.

Further, Mitsubishi Chemical has hitherto had manufacturing technologies, technical know-how, and manufacturing equipment for polyesters of an aromatic dicarboxylic acid and ethylene glycol or 1,4-butanediol, such as PET and PBT and thus

Table 1 Melting point(°C) of aliphatic polyesters polymerized from linear aliphatic dicarboxylic acids and linear aliphatic diols (Source: Saturated polyester handbook, K. Yumoto, The Nikkan Kogyo Shimbun, LTD)

| | | C number of aliphatic dicarboxylic acid | | | | | | | | |
|----------------------------|----|---|-----|-----|-----|----|----|----|----|----|
| | | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
| C number of aliphatic diol | 2 | 159 | -22 | 102 | -19 | 47 | 25 | 63 | 44 | 22 |
| | 3 | 66 | -25 | 43 | 35 | 36 | 41 | 47 | 46 | 49 |
| | 4 | 103 | -24 | 113 | 36 | 58 | 38 | | 49 | 64 |
| | 5 | 49 | -26 | 32 | 22 | 37 | 39 | 43 | 46 | 53 |
| | 6 | 70 | -48 | 52 | 28 | 55 | 52 | 61 | 52 | 65 |
| | 10 | 76 | 29 | 71 | 55 | 70 | 63 | 70 | 67 | 71 |
| | 20 | 88 | 67 | 86 | 77 | 85 | 82 | 86 | 34 | 87 |

Note: C number is “n” in HOOC-(CH₂)_n-COOH or HO-(CH₂)_n-OH

aimed for and succeeded in industrial production of PBS without using chain extenders.

1.3 Environmental Policy in Japan

As the countermeasure regarding climate change, the movement toward carbon neutrality has been activated. Responsive to this, high goals have been created all over the world, and policies thereof have been publicly announced. In Europe, in order to achieve the goals of the Paris Agreement toward realization of the carbon-neutral society, goals were set to make the amount of carbon dioxide emissions substantially zero in 2050, and it has been decided to achieve 55% reduction of carbon dioxide emissions in 2030 from the 1990 level. Also in the USA, after change of government in 2020, goals were created so as to make the amount of carbon dioxide emissions substantially zero in 2050, and in China, goals were declared so as to make the amount of carbon dioxide emissions substantially zero in 2060.

Meanwhile, in Japan, immediately after inauguration of the Prime Minister Suga in October 2020, he made the declaration of carbon neutrality and declared that in place of the previous goals for 80% reduction in greenhouse gases, it was aimed to achieve 100% reduction, namely to make the amount of carbon dioxide emissions substantially zero. In receiving the declaration of the Prime Minister Suga, in April 2021, Japan raised the goals for CO₂ reduction amount in 2030 from 26% to 46% in comparison with the 2013 year.

Further, the Japanese Government formulated the policy and the road map for achieving high goals toward the carbon-neutral society. Concretely, in May 2019, the Japanese Government formulated the circulation strategy for polymers (Fig. 2) and established milestones for reduce, reuse, recycle, and biomass plastics, respectively. In July 2020, a mandatory law for charging for plastic shopping bags that had

【Milestones】

| |
|--|
| <p><Reduce></p> <p>①25% reduction in emissions of one-way plastics by 2030</p> <p><Reuse/Recycling></p> <p>②Make design reusable/recyclable by 2025</p> <p>③Increase the ratio of reused or recycled containers and packaging to 60% by 2030.</p> <p>④100% effective use of used plastics by reusing or recycling by 2035.</p> <p><Recycling/Biomass plastics></p> <p>⑤Double the use of recycled materials by 2030.</p> <p>⑥Introduce about 2 million tons of biomass plastics by 2030.</p> |
|--|

Fig. 2 Plastics Recycling Resource Strategy and Milestones

been free of charge so far in supermarkets and convenience stores was enforced. In this law, marine biodegradable polymers or biomass polymers having a degree of biomass of 25% or more were excluded. As for the conversion of biomass into raw materials, the Japanese Government set highly challenged goals for introducing two million tons of bio-based plastics until 2030. Further, a bill regarding the “Plastic Resource Recycling Promotion Law” was passed in June 2021. According to this law, a guideline regarding the design for environment which manufacturers should serve was formulated, and retailers and restaurants, etc. were required to reduce the provision of disposable plastic products. This law focused on the “material” as the plastic and was aimed to promote the resource recycling at each stage of a life cycle from the designing and manufacturing stages of plastics until disposal.

2 BioPBS™ and FORZEAS™

2.1 Development of Bio-Based PBS by MCC

The progress of the development of PBS at Mitsubishi Chemical was summarized in Fig. 3. In 2003, Mitsubishi Chemical succeeded in the commercial production of the aliphatic polyester, PBS having an increased molecular weight in terms of a trademark “GS Pla™” without using a chain extender for the first time in the world. The “GS Pla™” in those days was manufactured for biodegradable applications in batch production using fossil-based succinic acid and 1,4-butanediol as the main raw materials [4].

Meanwhile, as described below, since around 2000, Mitsubishi Chemical has attempted to replace chemical raw materials by non-fossil-based resources and started to grapple the development of a group of chemical products satisfying high functionalization simultaneously with CO₂ reduction. In the “GS Pla™” business,

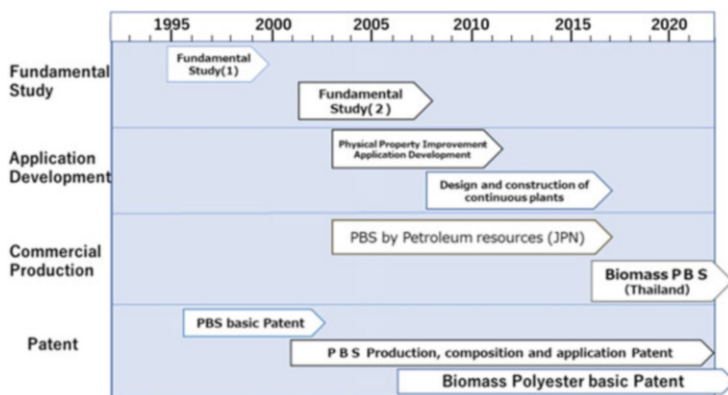


Fig. 3 History of PBS development at Mitsubishi Chemical Corporation

Mitsubishi Chemical proceeded to perform research and development aiming to launch the bio-based PBS business in which the raw material succinic acid is replaced by succinic acid easily derived from the biomass. However, at the time of commencement of the development, the bio-based succinic acid derived from the biomass resource contained a large quantity of impurities inherent to the biomass resource as the polymer raw material, and thus, PBS having a high molecular weight that could be possible for molding and exhibit sufficient mechanical strength was not produced at all. Even when molded, the resulting molded articles were inferior in designability such as poor color tone and many foreign matters. In order to solve these problems, it was essential to combine biochemistry for biomass conversion and chemical engineering for purification technology of the resulting polyester raw materials, with polymer science for high-molecular-weight polymerization using these raw materials and molding of the resulting polyester. Mitsubishi Chemical accomplished conversion technology into a monomer for polyester with economic rationality and biodegradability control technology by biochemistry, efficient purification process with economic rationality by chemical engineering, and molecular weight-increasing technology using bio-based monomers containing specific impurities and molding technology by polymer science [5]. Afterward, Mitsubishi Chemical granted a license of technology to J/V established in 2011 together with Petroleum Authority of Thailand (PTT), designed continuous large-sized full-scale commercial plant (20k Ton/Y) for the first time in the world, and commenced the production of the bio-based PBS (trademark: BioPBS™) made of bio-based succinic acid and fossil-based 1,4-butanediol at PTTMCC Biochem Company Limited since 2017. At the time of 2021, the raw material 1,4-butanediol is derived from petroleum, but Mitsubishi Chemical plans to change it to a bio-based raw material in the near future. When this is realized, the business of all bio-based completely degradable polymers will be operated.

2.2 Comparison of Biodegradable Polymers

BioPBS™ is a polymer that is not only biodegradable but also bio-based. Polymers called as a biopolymer or green polymer are needed to be arranged in terms of two points including the viewpoint of biodegradation and whether the raw material is bio-based or fossil-based (Fig. 4). BioPBS™, PLA, and PHB are not only bio-based but also biodegradable, and bio polycarbonate (e.g., DURABIO™), bio polyethylene, bio nylon, and bioPET are bio-based but non-biodegradable polymers. Further, PCL and PBAT are fossil-based biodegradable polymers. Almost all of polymers which are currently put into practical use are fossil-based and non-biodegradable polymers.

Table 2 listed basic physical properties such as thermal properties and mechanical properties of biodegradable polymers and biomass polymers which were put into practical use. PHB and PLA are bio-based polymers and hard-type polymers as in PET, polystyrene, etc., and PCL, PBS, and PBSA are soft-type polymers having

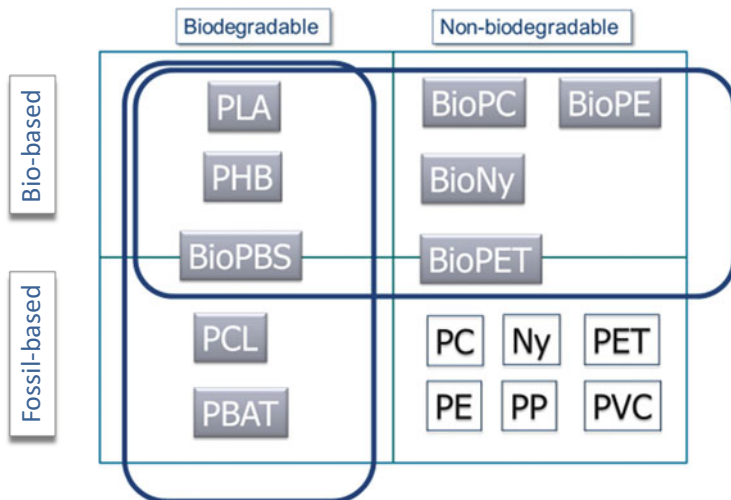


Fig. 4 Classification of biodegradable plastics and bio-based plastics

physical properties analogous to high density polyethylene (HDPE), low density polyethylene (LDPE), etc. PBAT is a soft biodegradable polymer having a lower elastic modulus. Because there are a few of different types of commercialized biodegradable polymers, for practical implementation of biodegradable polymers, in order to achieve necessary requirement characteristics, these polymers are often blended and used so as to have optimal properties and optimal biodegradation rate through complementary use or blending. PBS and PBSA are biodegradable polymers having heat resistance and mechanical properties close to LDPE. The grades and basic physical properties of BioPBS™ will be described in detail in another chapter.

2.3 Biomass Conversion of Raw Material

The chemical industry has grown so far in a way that provides safety and security to the society, with the objective of providing technologies for pursuing convenience and comfortability of people through high functionalization of products, as well as achieving energy conservation through ruggedization and weight reduction. Meanwhile, it has become an era such that countermeasures against the climate change with an increase in atmospheric CO₂ emissions, and the depletion issue of fossil fuels, in recent years, as well as environmental loads on a global scale, such as a waste plastic issue and a recent marine plastic issue must be taken.

However, since most of the current chemical products have largely depended on petroleum as a fossil resource, the chemical industry is required by society to create technologies that eliminate petroleum as a resource. Among such societal demands,

Table 2 Properties of biodegradable plastics and biomass plastics

| | PHB | PHB/V | PLA | PVA | PCL | PBS | PBSA | PES | PBAT | LLDPE |
|------------------------------|-------|-------|-------|-----|-----|-----|------|-----|------|-------|
| Glass transition temperature | 4 | | 59 | 74 | -60 | -22 | -36 | -11 | -30 | <-70 |
| Melting point | 180 | 151 | 179 | 210 | 60 | 115 | 84 | 100 | 115 | 108 |
| Flexural modulus | 2,600 | 1,800 | 3,500 | - | 280 | 640 | 250 | 750 | - | 150 |
| Tensile strength | 26 | 28 | 55 | 1 | 61 | 36 | 24 | 25 | 25 | 18 |
| Tensile elongation at break | 1 | 16 | 2 | 2 | 730 | 210 | 380 | 500 | 620 | 700 |
| Izod impact strength | 12 | 161 | 3 | 13 | NB | 10 | 47 | 186 | 45 | 50 |

one of the directions of the technical development which the chemical industry should accomplish in the future is to develop products or systems with life cycle assessment for reducing the amount of CO₂ emissions, in addition to high functionality and high durability of chemical products to be manufactured.

As the chemical process solving the CO₂ reduction issue is on a global scale, a process for utilization of a biomass resource or CO₂ into a chemical product has been proposed, in addition to accomplishment of an industrial process technology of further energy conservation type [6]. In the polymer industry, an interest in biomass conversion of raw materials is also high, and a large number of researches have been made so far. However, in the polymer manufacture, polymers aiming for conversion of raw material into biomass should be highly versatile polymers that could be mass-produced from that purpose. Further, in general, so far as biomass resources are concerned, in case of C6 sugar, it is a raw material represented by C₆(H₂O)₆ with inferior energy to petroleum resources, and thus, it is important to select the target bio-based polymer. From this viewpoint, the polymer derived from the biomass is desirably a polymer using a raw material relatively easily derived from biomass resources, or a polymer using a raw material with oxygen atoms derived directly from the biomass capable of reducing the energy consumption during manufacture.

Meanwhile, in general, the biomass raw material is a compound rich in reactivity and is characterized such that impurities are more contained, as compared to fossil resource-derived raw materials. In particular, the polymer raw material is required to be highly purified as compared to general chemical products, and thus, its conversion reactions with high reaction selectivity capable of reducing a load of the purification process of a crude product derived from the biomass are required. Moreover, the versatile polymer is required to be a polymer with high economic rationality because it is a replacement for existing polymers, and thus, in the derivatization reaction from the biomass, it is important that the production process has economic rationality with a reduced number of reaction steps and is of an energy conservation type.

As a promising candidate for such a polymer raw material, there are exemplified succinic acid and 1,4-butanediol as the raw materials of PBS as taken up in this article and besides, furan derivatives, isosorbide, and ethanol-derived ethylene or ethanediol, a part of which is commercially produced.

2.4 Development of Biodegradable Polymer Compound FORZEAS™

FORZEAS™ is a biodegradable polymer compound using BioPBS™.

FORZEAS™ is an earth-friendly material which contributes to reduction of use of limited raw materials and also contributes to reduction of plastic wastes through degradation into water and carbon dioxide by potency of microorganism in the natural world. FORZEAS™ is based on BioPBS™ making the best use of high heat resistance and high compatibility of BioPBS™ and has a performance which

cannot be exhibited by a single body, through compounding with other biodegradable polymer. FORZEAS™ is provided with product grades according to various applications such as injection molding, sheet, inflation film, straw, lamination, and zipper and has characteristics friendly to the environment so as to symbolize “KAITEKI” as the philosophy of Mitsubishi Chemical Holding.

3 Characteristic Feature of BioPBS

3.1 *BioPBS™ (Outline of Bio-Based PBS)*

BioPBS™ is a polyester of environmental harmony type developed by Mitsubishi Chemical for the purposes of breaking away from the conventional unidirectional chemical system of production, consumption and exhaustion from fossil resources unevenly distributed on the earth and constructing a cycling-based social system. BioPBS™ is a polyester which using a renewable biomass resource as the chemical raw material, cuts off the depletion issue of fossil resources and diversifies the raw material resources, and which is expected to construct a carbon cycling-based chemical system largely contributing to the CO₂ emission reduction by immobilizing CO₂ emitted by burning or degradation of used plastics with the growth of plants using solar energy (Fig. 5).

Although this chemical system itself is one proposed from a long time, it is hardly accepted by the current market merely when the chemical raw material is simply replaced from the fossil-based resource by the bio-based resource raw material, and it is the actual situation that its market value is low unless it is a technology capable of reducing the costs of plastics or a technology capable of enhancing the functions of plastics by use of the bio-based raw material, or a technology in which the manufacture cannot be performed without using the biomass raw material. From that viewpoint, BioPBS™ has characteristic features that it is able to be molded into various molded products, has both practical strength and durability, and has a function such that it is excellent in degradability under soil as compared to the fossil resource-derived PBS [5] (Fig. 6). In this way, BioPBS™ aims to achieve the carbon cycling of chemical raw materials and is expected as a polymer capable of achieving both high functionalization and CO₂ reduction at the same time, particularly, the function of its biodegradability has got high evaluation in food applications and agricultural multifilm applications.

3.2 *Manufacturing Technology*

PBS having an increased molecular weight exhibits flexible and supple mechanical ~~physical~~ properties similar to polyethylene and is expected to be adopted for various applications [7]. However, as described above, the difficulty of manufacture of

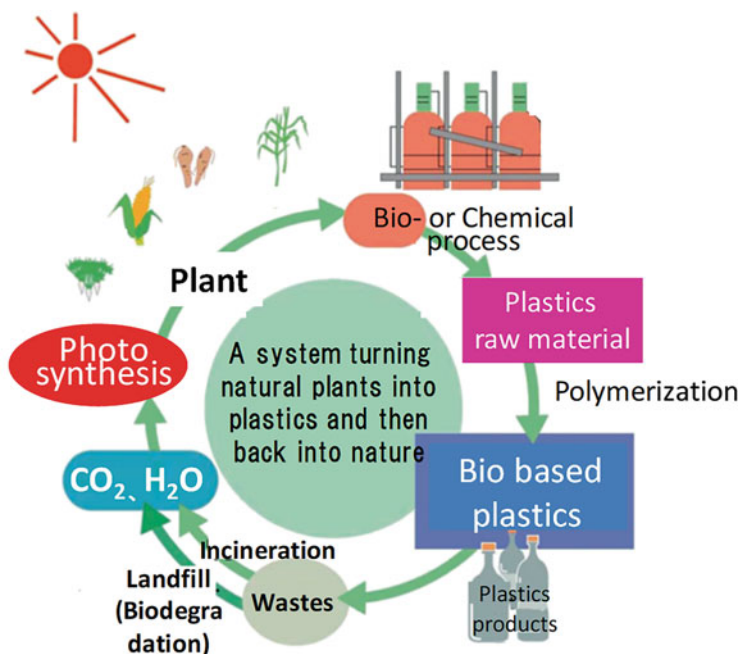


Fig. 5 Concept of environmentally benign bio-based plastics

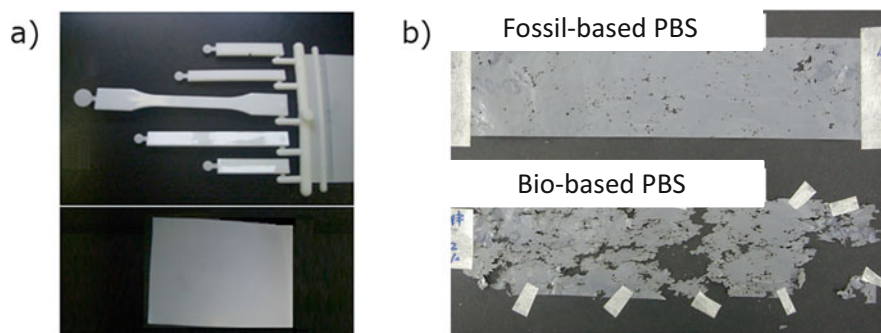


Fig. 6 Bio-based PBS. (a) Various molded products. (b) Difference in the rate of change in film shape from fossil-based PBS after degradation in soil

high-molecular-weight PBS having such practical mechanical properties is caused due to the point that in order to reveal practical mechanical properties, the molecular weight is needed to be raised as compared to aromatic polyesters such as PET and the point that the heat stability of PBS is low. As shown in Fig. 7, in the manufacture of PBS, it is a manufacturing method with economic rationality to adopt a melt polymerization method consisting of a two-stage process of an esterification step of condensation reaction between succinic acid and 1,4-butanediol and a

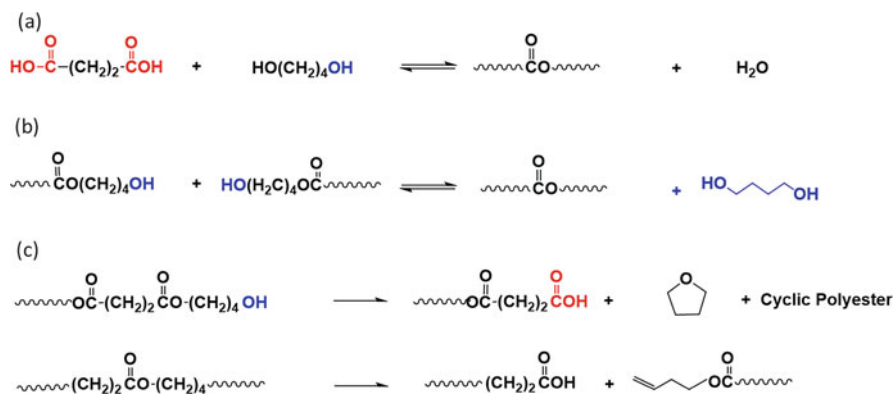


Fig. 7 General scheme of PBS synthesis. (a) Esterification. (b) Transesterification. (c) Decompositions during polymerization

transesterification step under reduced pressure. However, the melt polycondensation reaction of PBS involves such characteristic features that simultaneously with the reaction of raising the molecular weight of PBS, degradation reaction of PBS concurs, for example, tetrahydrofuran and cyclic polyesters are by-produced due to backbiting of polymer terminals, and degradation reaction of PBS such as production of a carboxy group and an olefin group concurs due to thermal degradation in the ester site. In order to control such degradation reaction, a process technology is necessary to produce PBS by lowering the polymerization temperature or shortening the polymerization time. With respect to the method for increasing the molecular weight of PBS, detailed investigations such as an influence of the kind and addition amount of a used polymerization catalyst [8] and reaction rate analysis at the time of polycondensation reaction time [9] were made. Besides, new manufacturing processes such as a polycondensation method [10] and a solid phase polymerization method [11], in which distillation efficiency of the distillate was enhanced using an organic solvent, were also proposed. However, the PBS manufacturing method with economic rationality was not resulted even when adopting merely these technologies, and before Mitsubishi Chemical commercialized “GS Pla®” and “BioPBS™,” only PBS using a chain extender had been manufactured [12, 13]. In contrast, Mitsubishi Chemical designed the large-sized full-scale commercial plant of not only optimizing a used catalyst and its addition amount but also enhancing the distillation efficiency of a distillate by-produced during the polycondensation reaction and introduced into PTT MCC Biochem Company Limited. Owing to introduction of this continuous production process, Mitsubishi Chemical has successfully accomplished the manufacturing technology of bioPBS having high molecular weight, high practical strength and durability, and excellent designability.

3.3 Basic Characteristics of PBS

Table 3 shows the main physical properties of BioPBS™. For comparison, those of PE, PP, PLA, and PS are also shown in this table. “BioPBS™” has two basic grades: FZ Series (PBS) and FD Series (PBSA). The FZ Series have the melting point analogous to PE, but are inferior in transparency. This is closely related to the crystallinity of PBS. The FD Series are the copolymer with adipic acid and the crystallinity is controlled by optimization of a nucleating agent to improve the transparency and the flexibility.

PLA whose market development is energetically carried out is a rigid material and has characteristics significantly different from the flexible PBS. These two types of materials are spread in a different application field from each other and also may be thought to be used as a polymer blend aiming at complementation of mechanical properties and thermal characteristics. Table 4 shows comparison in characteristics between PBS and PLA. In addition, Fig. 8 shows an example of the morphology in the PBS/PLA blend. Finely dispersed sea-island structure of PBS and PLA with the order of several hundred nm is investigated here. PBS used here is the product formerly manufactured in Japan by Mitsubishi Chemical (hereinafter referred to as

Table 3 Basic properties of various polymers

| | | PBS | PBSA | LLDPE | PP | PLA | PS |
|---|-------------------|---------|------|-------|------|------|------|
| Density | g/cm ³ | 1.26 | 1.24 | 0.92 | 0.90 | 1.26 | 1.05 |
| Glass transition temperature | °C | – 22 | –36 | <–70 | –10 | 59 | 100 |
| Melting point | °C | 115 | 84 | 108 | 165 | 179 | - |
| Tensile strength | MPa | 36 | 24 | 18 | 30 | 55 | 40 |
| Tensile elongation at break | % | 210 | 380 | 700 | 700 | 2 | 2 |
| Flexural modulus | MPa | 650 | 250 | 150 | 1300 | 3500 | 3150 |
| Notched Izod impact strength(23°C) | kJ/m ² | 10 | 47 | 50 | 3 | 3 | 2 |
| Heat deflection temperature (0.45 MPa) | °C | 95 | 63 | 45 | 115 | 59 | 100 |

Table 4 Comparison of characteristics between PBS and PLA

| | PBS | PLA |
|---------------------------------------|-------------|-------------|
| Flexibility | Flexible | Rigid |
| Impact strength | Higher | Lower |
| Transparency | Translucent | Transparent |
| Crystallization speed | Higher | Lower |
| Heat resistance | Higher | Lower |
| Heat-seal temp. | Lower | Higher |
| Process temp. | Lower | Higher |
| Biodegradability(30°C/50% RH in soil) | Faster | Slower |

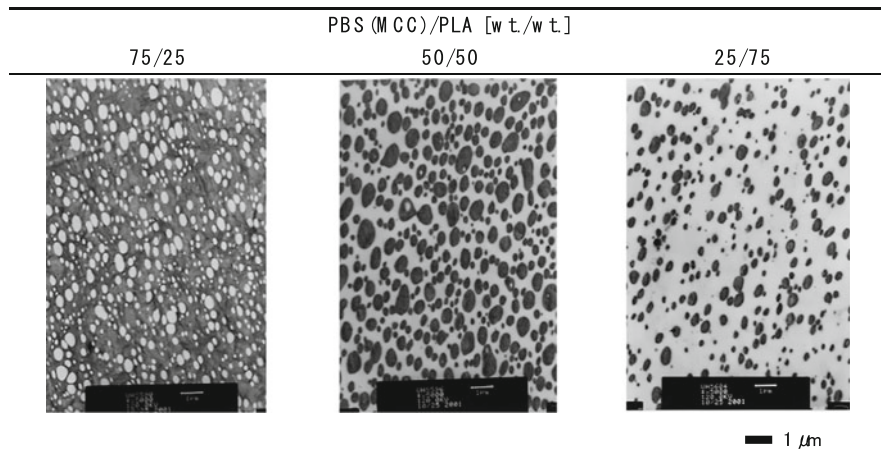


Fig. 8 Morphology of PBS/PLA blend

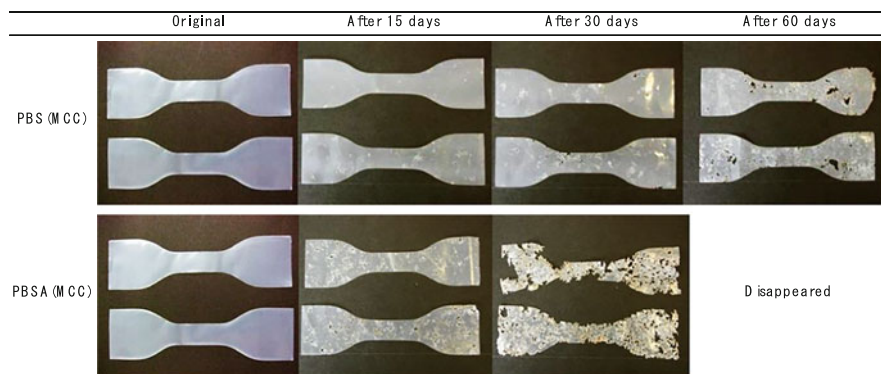


Fig. 9 Field test in soil. Specimen: 40 μm thickness blown film. Location: Field of Yokohama R&D Center, Mitsubishi Chemical Corporation. Conditions: Not controlled (Natural climate)

PBS(MCC)). Furthermore, blending with PBAT is often carried out to improve the film strength.

3.4 Biodegradability and Certification in Various Regions

PBS and PBSA have relatively good biodegradability. Figures 9 and 10 show the field test results of the biodegradability carried out in the farm field at Yokohama R&D Center of Mitsubishi Chemical. As indicated in these figures, PBS and PBSA are relatively quickly degraded (precisely disintegrated) even in the environment where PLA or PBAT is not substantially degraded. The reason of this difference can

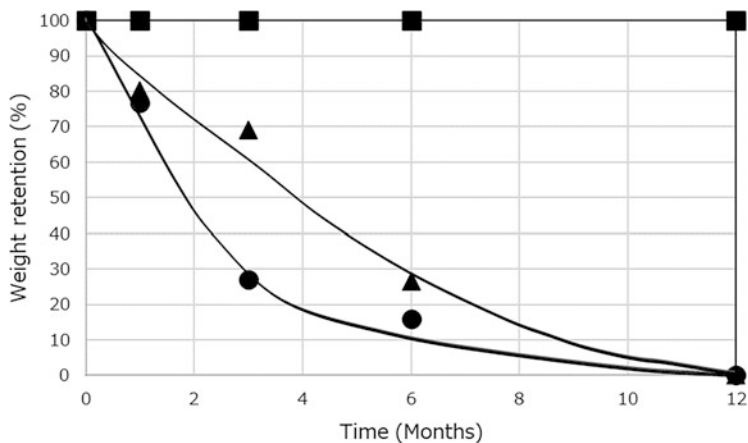


Fig. 10 Disintegration test of 200 μm thickness sheet in soil under 30°C and 50%RH. Filled triangle: PBS(MCC). Filled circle: PBSA(MCC). Filled square: PLA

be attributed to the low glass transition temperature and the chemical structure of PBS and PBSA. As the glass transition temperature of PBS and PBSA is not higher than 0°C, the amorphous region is in the rubbery state with high mobility in the usual environment, and in addition, the basic structure of PBA and PBSA is only composed of aliphatic. In general, lowering the molecular weight by the polymer chain scission due to the non-biological hydrolysis and the biological hydrolysis by microorganisms is necessary for the biodegradation and in order to proceed the hydrolysis more rapidly higher mobility of the polymer chains in the amorphous region is preferable. On the other hand, as the amorphous region of PLA is in the glassy state at room temperature due to the glass transition temperature of about 60°C, PLA is disadvantageous for hydrolysis. Meanwhile, it is known that when the temperature rises to about 60°C, PLA is quickly biodegraded. A polymer whose molecular weight is lowered to some level is mineralized into water and carbon dioxide by microorganisms, namely biodegraded. Microorganisms capable of secreting the enzyme degrading the PBS and PBSA are considered to be generally contained in the soil. Furthermore, PBSA is biodegraded faster than PBS due to the lower degree of crystallization and the faster hydrolysis of PBSA. A schematic view of biodegradation process is shown in Fig. 11.

Thanks to this characteristic feature, each BioPBS™ already acquires the biodegradability certification in various regions as shown in Table 5. Fig. 12 shows an example of the test results carried out at OWS in acquiring the certification. All of the disintegration test, the biodegradability test, the germination test, and the earthworm test demonstrate that BioPBS™ has performances satisfactory in acquiring each certification. Incidentally, as biodegradation rate of the FZ Series is lower than that of the FD Series due to the higher crystallinity, the FZ Series are not able to acquire the “OK compost HOME” certification, but as shown in Fig. 10, if the conditions are

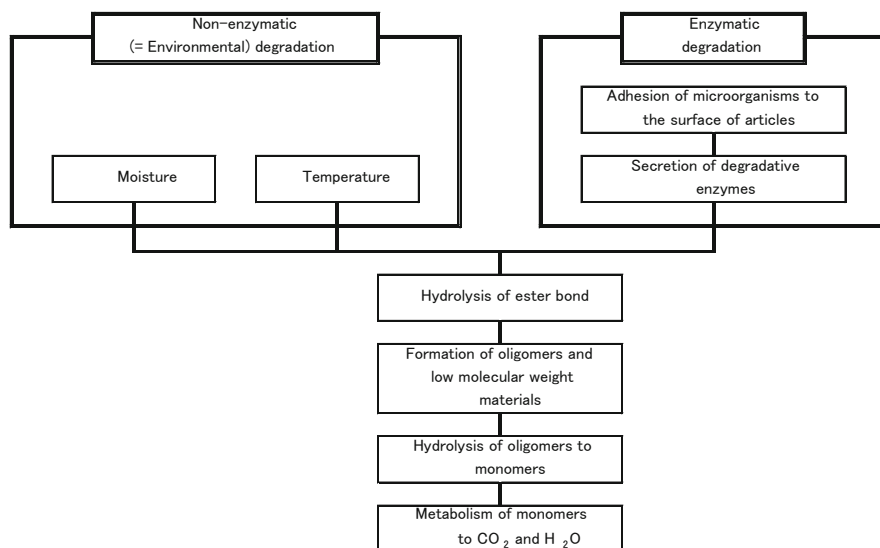


Fig. 11 Schematic view of biodegradation process

met, the FZ Series are also biodegraded in the environment even at ambient circumstances.

3.5 Certification of Bio-Content

As mentioned already, succinic acid that is one of the main raw materials of BioPBS™ is bio-based. Furthermore, we have a plan to use the bio-based 1,4-butanediol that is another main raw material in the future. Bio-content of BioPBS™ at the present stage is shown in Table 6. In addition, the comparison in the major physical properties between the conventional fossil-based PBS and the current bio-based PBS is shown in Table 7. As indicated in this table, physical properties of the bio-based PBS are revealed to be equal to the fossil-based PBS.

3.6 Food Contact Certification

BioPBS™ also acquires the certification of food contact certifications in various regions. As shown in Table 8, the FZ Series are certified in Japan, EU, USA, and China, and the FD Series are certified in Japan, EU, and USA. The FZ Series having a higher melting point can be used under higher temperature conditions.

Table 5 Biodegradable certifications of BioPBS™

| | Japan | | EU | | US | | Australia | |
|--------------|-----------|-----------------------|------------------------|-----------------|-----------------------|-----------------------|-----------------------|------------------|
| | GreenPla | OK compost | OK compost | OK compost HOME | Compostable | Compostable | Compostable | Home compostable |
| | JBPA | TUV Austria | | | BPI | ABA | | |
| FZ91 (PBS) | Certified | Certified (max.69 µm) | – | | Certified (max.68 µm) | Certified (max.74 µm) | – | |
| FZ71 (PBS) | Certified | Certified (max.74 µm) | – | | Certified (max.74 µm) | Certified (max.74 µm) | – | |
| FD92 (PBBSA) | Certified | Certified (max.87 µm) | Certified (max.502 µm) | | Certified (max.87 µm) | Certified (max.87 µm) | Certified (max.85 µm) | |

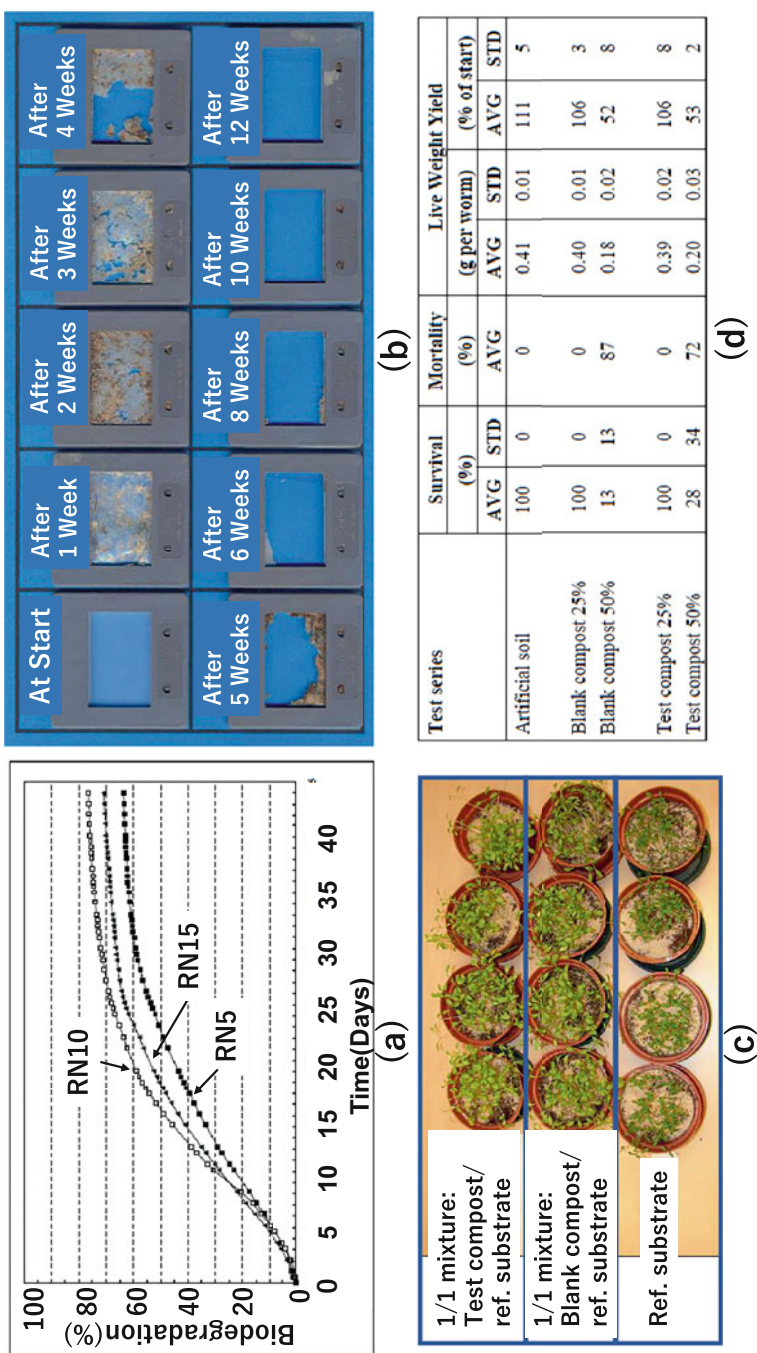


Fig. 12 Biodegradability test of PBS by OWS. (a) Aerobic biodegradation under $58 \pm 2^\circ\text{C}$. Average biodegradation percentage relative to cellulose was 97.1%. (b) Disintegration of PBS film under $58 \pm 2^\circ\text{C}$. (c) Overview of the growth of cross plants at the end of the test. From top to bottom: PBS compost/reference substrate = 1/1 blank compost/reference substrate = 1/1 reference substrate. (d) Earthworm, acute toxicity test on compost residuals of PBS

Table 6 Bio-content of BioPBS™

| | EU DIN CERTCO |
|-------------|---------------|
| FZ91 (PBS) | 51% |
| FZ71 (PBS) | 51% |
| FD92 (PBSA) | 35% |

Table 7 Comparison of bio-based PBS and fossil-based PBS

| | | Bio-based | Fossil-based |
|---------------------------------------|-------------------|-----------|--------------|
| Density | g/cm ³ | 1.26 | 1.26 |
| Glass transition temperature | °C | -22 | -22 |
| Melting point | °C | 115 | 115 |
| Tensile strength | MPa | 36 | 30 |
| Tensile elongation at break | % | 210 | 160 |
| Flexural modulus | MPa | 650 | 640 |
| Notched Izod impact strength(23°C) | kJ/m ² | 10 | 7 |
| Heat deflection temperature(0.45 MPa) | °C | 95 | 93 |

Table 8 Food contact certifications of BioPBS™

| | Japan JHOSPA | EU No.10/2011 | US FCN | China GB4806 |
|-------------|------------------------|---------------|---------------|------------------------|
| FZ91 (PBS) | Certified ^a | OM6 | No.1817 (B-H) | Certified ^b |
| FZ71 (PBS) | Certified ^a | OM6 | No.1817 (B-H) | Certified ^b |
| FD92 (PBSA) | Certified ^c | OM2 | No.1818 (C-G) | - |

^a 100°C or less. Inapplicable to dairy products

^b 100°C or less

^c 65.6°C or less. Inapplicable to alcoholic products and dairy products

3.7 Molding Processability

The DSC results of PBS and PLA are shown in Fig. 13. Since PBS has a relatively fast crystallization rate, it is applicable to almost all of the molding methods (e.g., blown film fabrication, extrusion molding, sheet molding, injection molding, and vacuum molding) which are used for current plastic materials and can be widely chosen according to the application. Examples of representative molding conditions are shown in Tables 9, 10, and 11.

3.8 Storage Stability

As for the biodegradable polymer, the hydrolysis gradually proceeds in the hygroscopic state, and the mechanical properties are lowered. Thus, it is important to make an estimate of storability of the products using the biodegradable polymer. Though there are several thoughts about the estimate of storability, an example of the estimate will be given below.

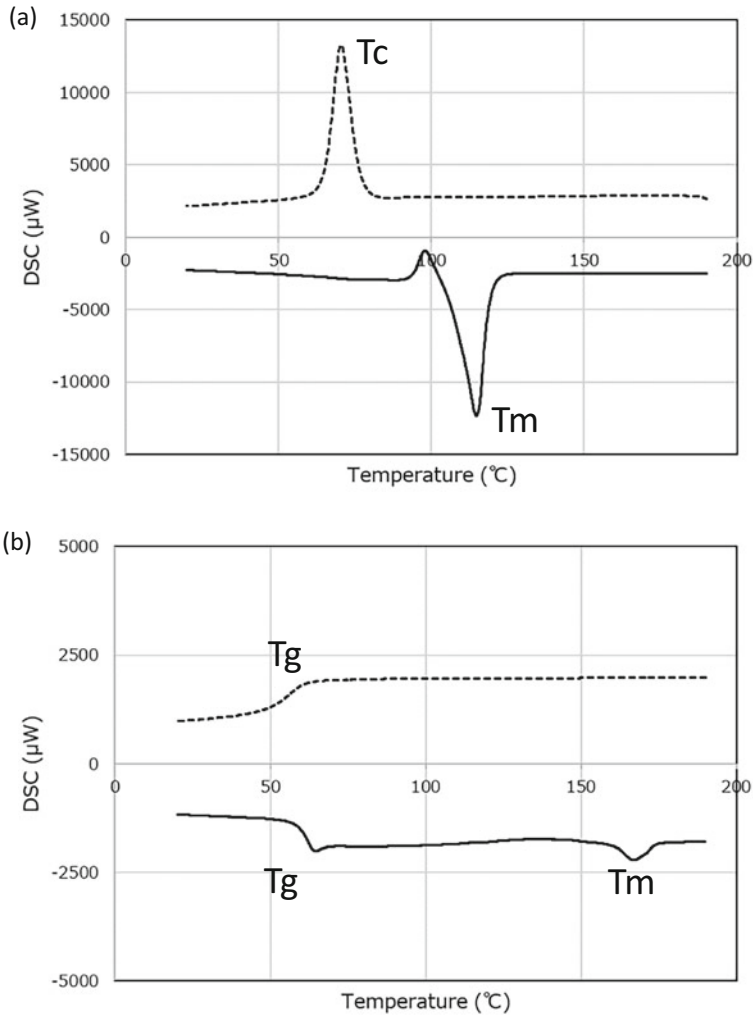


Fig. 13 DSC diagrams of (a) PBS and (b) PLA. —: Heating (10°C/min). ---: Cooling (10°C/min)

Figure 14 schematically shows the relation between the solution viscosity IV and the tensile elongation at break when taking a film as an example. For example, if the performance as the product cannot be satisfied when reaching the arrowed tensile elongation at break, a threshold value T_h of IV of the product can be estimated. Meanwhile, assuming that the change of IV with time under a certain temperature and humidity is like Fig. 15, a period until IV reaches T_h is an estimated usable period (SL) as the product. The results obtained by these experiments under several conditions are shown in Fig. 16. In this case it is presumed that the product quality is maintained for about 300 days in the environment at 30°C and 50% RH. Actually, as not only the temperature and the humidity change, but also the physical properties

Table 9 Example of blown film fabrication conditions

| | | FZ91 | | FD92 |
|----------------|----------|------|-----|------|
| Temperature | (°C) | C1 | 150 | 140 |
| | | C2 | 160 | 150 |
| | | C3 | 160 | 150 |
| | | H | 160 | 150 |
| | | D | 160 | 150 |
| Die lip gap | (mm) | | 1 | 1 |
| Screw speed | (rpm) | | 40 | 40 |
| Take-up speed | (m/min.) | | 10 | 10 |
| Blow-up ratio | (-) | | 2.5 | 2.5 |
| Film width | (mm) | | 240 | 240 |
| Film thickness | (μm) | | 30 | 30 |

Table 10 Example of extrusion coating conditions

| | | FZ91 | |
|---------------|----------|-------------------|-------|
| Temperature | (°C) | C1 | 230 |
| | | C2 | 245 |
| | | C3 | 245 |
| | | D1 | 245 |
| | | Roll ^a | 20–30 |
| Take-up speed | (m/min.) | | 100 |

^a Semi-matte finish chill roll

Table 11 Example of injection molding conditions

| FZ71 | | | |
|--------------------|--------|----|-----|
| Temperature | (°C) | NH | 180 |
| | | H1 | 180 |
| | | H2 | 180 |
| | | H3 | 180 |
| Mold temperature | (°C) | | 40 |
| Screw speed | (rpm) | | 70 |
| Injection pressure | (MPa) | | 65 |
| Hold pressure | (MPa) | | 35 |
| Injection time | (sec.) | | 15 |
| Cooling time | (sec.) | | 30 |

adopted as the threshold value and the threshold value thereof vary according to the product form, the situation is very complex. However, this kind of consideration is important in order to image the storage period of the product.

3.9 Examples of Applications

Application examples of PBS and PBSA will be given below.

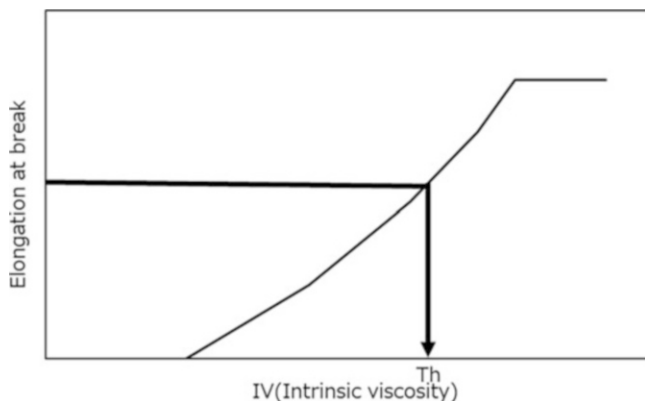


Fig. 14 Schematic view to determine the threshold value(T_h)

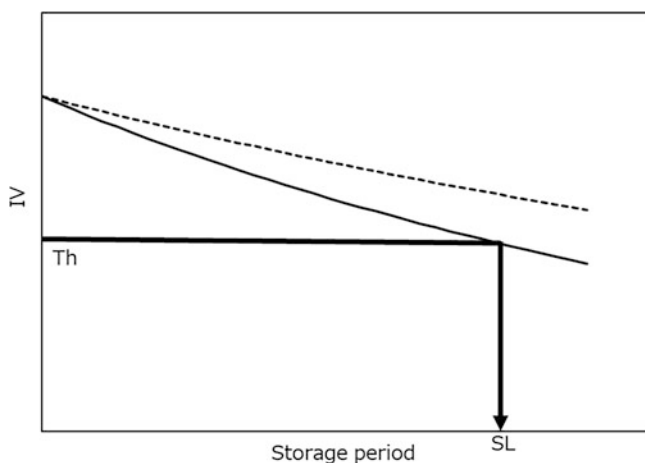


Fig. 15 Schematic view to determine the shelf life(SL). –: Condition A. --: Condition B

3.9.1 Food Contact Application

As mentioned already, BioPBS™ acquires the food contact certifications in various regions. In addition, PBS has good heat-sealing properties (Fig. 17) and is frequently used as the sealant for food packaging applications due to these characteristics. A representative example thereof is a paper coating application. Though PE is usually used for the inner surface of the paper cup for the purpose of water resistance and sealant in the joint part, PE must be separated in the recycling process of paper cup. Meanwhile, regarding the PBS coated paper cup PBS is expected to be hydrolyzed under alkaline treatment conditions of the paper recycling process with maintaining the properties of water resistance and high heat sealability. As the result of these PBS properties, the load of waste disposal in the recycling process can be expected to be

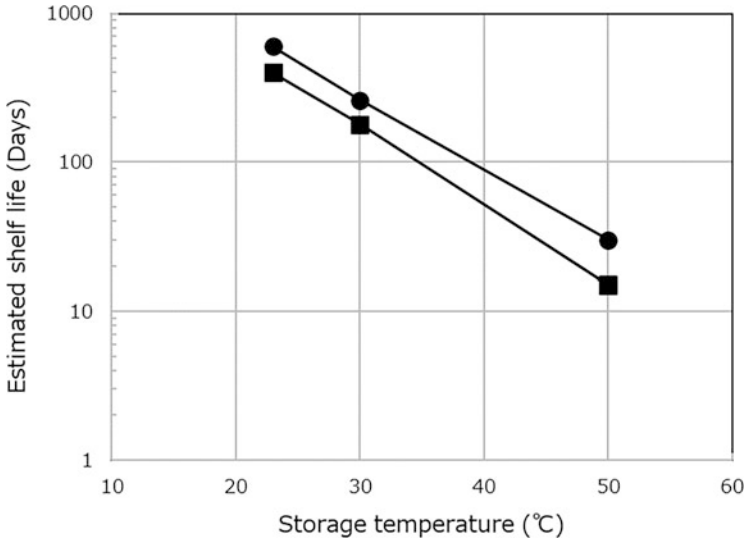


Fig. 16 Dependence of estimated shelf life of PBS film on the storage conditions. Filled circle: 50%RH. Filled square: 90%RH

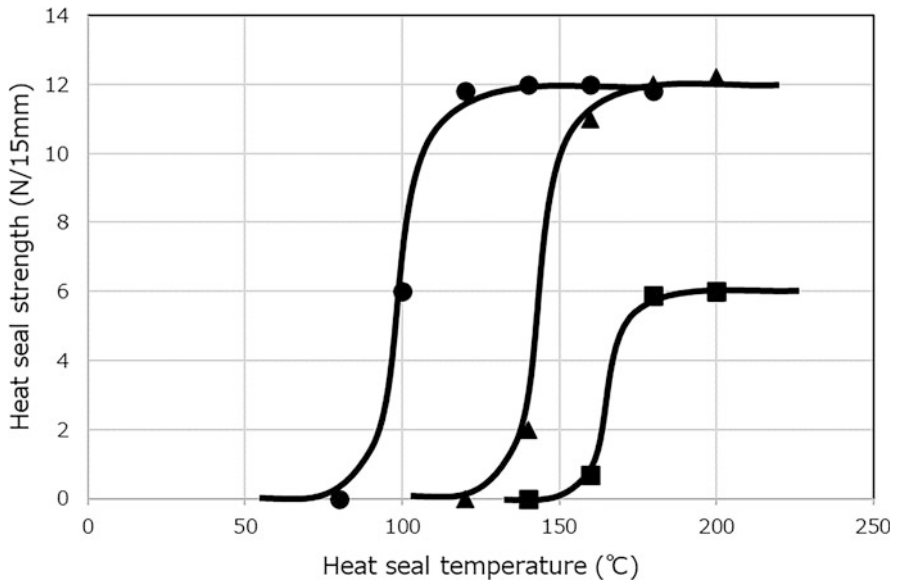


Fig. 17 Dependence of heat-seal strength on heat-seal temperature [Heat-seal conditions]. Film thickness: 20 μ m/Pressure: 0.2 MPa/Time: 2 s/Seal width: 15 mm. Filled triangle: PBS. Filled circle: PBSA. Filled square: LLDPE

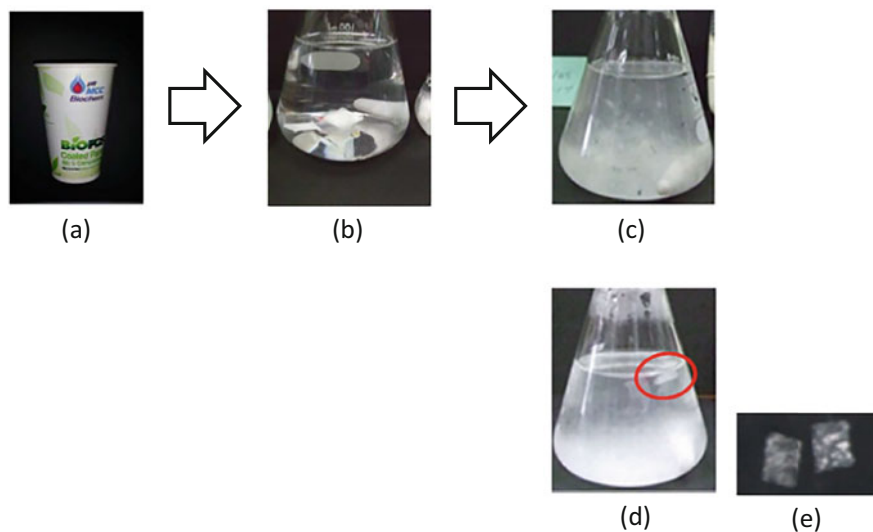


Fig. 18 Recyclability test of paper cup. (a) PBS or PE coated paper cup. (b) Paper cup pieces were stirred in 70°C/pH 13 water for 30 min. (c) No residue was observed for PBS coated paper cup after treatment. (d) PE film residues (e) were observed for PE coated paper cup after treatment



Fig. 19 Composting test of PBS coated paper cup. (a) PBS coated paper cups were mixed with the substrate in the composting machine. (b) Almost no residues of paper cup were observed after 24 h treatment

reduced (Fig. 18). Recently Mitsubishi Chemical is jointly carrying out the demonstration experiments with the professional football club in Japan. The PBS coated paper cups provided in the football stadium are collected and composted together with the other food wastes and the obtained composts are used to cultivate the vegetables in the field of the neighborhood (Fig. 19). Moreover, utilizing the high heat resistance of PBS compared to PLA (Fig. 20), Mitsubishi Chemical prepares some grades for the lid of paper cup, tableware, and straw in combination with the paper cup.

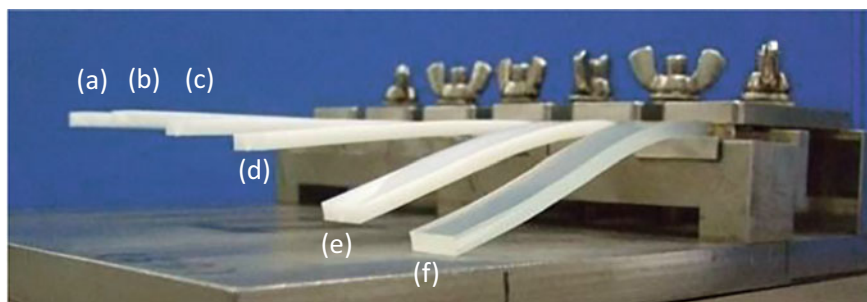


Fig. 20 Heat sag test under 80°C for 1 h of PBS/PLA blend. (a) PBS(MCC)/PLA = 100/0 wt./wt. (b) PBS(MCC)/PLA = 70/30 wt./wt. (c) PBS(MCC)/PLA = 40/60 wt./wt. (d) PBS(MCC)/PLA = 25/75 wt./wt. (e) PBS(MCC)/PLA = 10/90 wt./wt. (f) PBS(MCC)/PLA = 0/100 wt./wt

Table 12 Gas transmission rate of PBS

| | O ₂ (cc/atm·m ² ·24 h) | CO ₂ (cc/atm·m ² ·24 h) | H ₂ O (g/m ² ·24 h) |
|------|--|---|---|
| PBS | 770 | 6,600 | 620 |
| PLA | 1,900 | 4,000 | 680 |
| LDPE | 8,850 | 55,500 | 26 |
| PET | 75 | 525 | 34 |

Specimen: 20 μm thickness blown film (converted)

Test conditions: O₂: 23°C, 50% RH

CO₂: 23°C, 0% RH

H₂O: 40°C, 90% RH

Table 13 Aroma retaining test

| | d-limonene (g/m ² ·24 h) |
|------|-------------------------------------|
| PBS | 120 |
| LDPE | 710 |

Specimen: 20 μm thickness blown film

Since the oxygen barrier property of PBS is low as shown in Table 12, PBS itself is unsuitable for food packages requiring the storage for a certain period of time, but the case where PBS is used as the sealant of cellulose or paper with excellent gas barrier properties is increasing. Moreover, Mitsubishi Chemical is investigating the multilayered sheet for the total solutions of biodegradable packaging using PBS-based compound as the inner and the outer layer and biodegradable polymer with excellent oxygen barrier property as the middle layer.

As PBS is a type of polyesters, PBS indicates excellent printability and aroma retaining property like terpene compared to PE (Table 13). In addition, as the demand to the marine biodegradability and the home compostability is increasing, Mitsubishi Chemical is developing some grades having such functions (Figs. 21 and 22). In the future, Mitsubishi Chemical is going to expand such highly functionalized products.



Fig. 21 Disintegration test under 28°C (referring to ISO 20200) by OWS. Specimen: 111 μm thickness film of developing grade

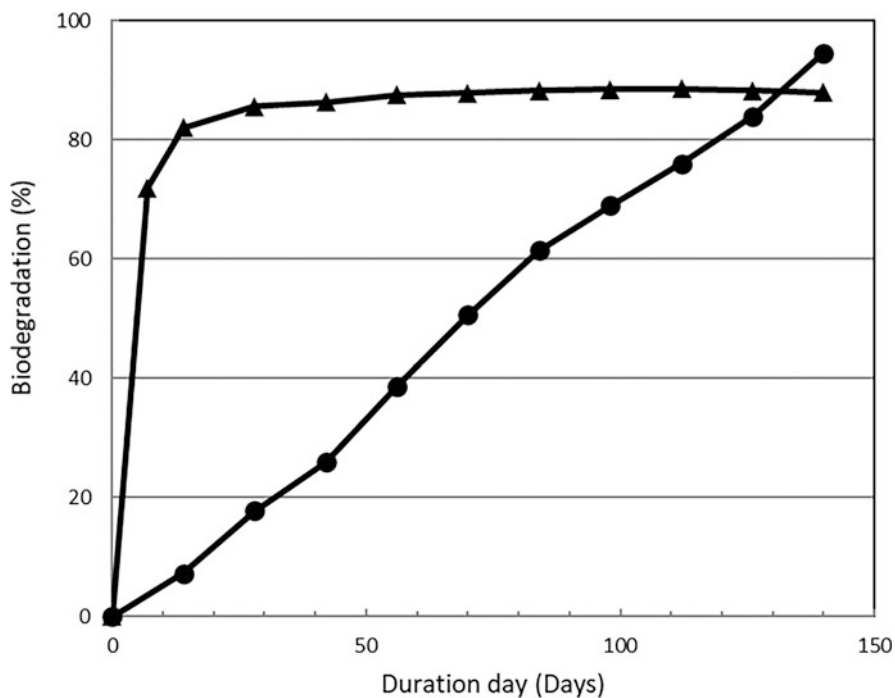


Fig. 22 Marine degradation test (referring to ASTM D6691) by OWS. Filled circle: Developing grade. 95% degradation after 140 days. Filled triangle: Cellulose as reference. 88% degradation after 140 days

3.9.2 Agricultural Mulching Film

For mulching films covering the farmland aiming at growth promotion of crops or weed prevention, PE films are generally used. However, prodigious labor to remove the films from the farmland after harvesting crops is needed. Sometimes roots or stems twining around the films make the works more tough. Furthermore, a lot of time and cost are taken for the waste disposal of the collected films. Though the biodegradable film is higher in cost than the PE film, farmers who have once used the biodegradable mulching film use the biodegradable mulching film repeatedly in the next season due to the convenience (Fig. 23). In recent years, films in which the biodegradability/durability, strength, color, moisture permeability, etc. are adjusted according to the area or crop have been developed and are expected to be more widely used in the future. In addition, as shown in Fig. 24, enzymes capable of quickly degrading the biodegradable polymer are also available, and Mitsubishi Chemical is pursuing further convenience of biodegradability by advancing development of such technology.

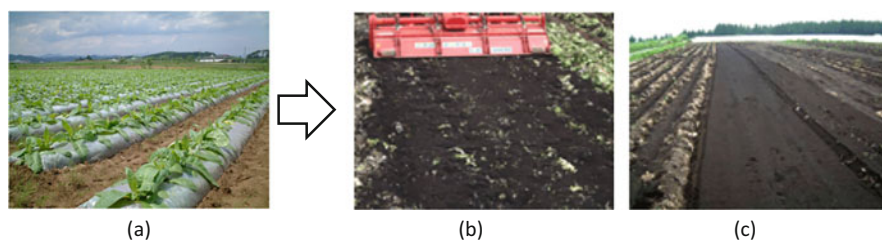


Fig. 23 Agricultural mulching film (a) before and (b), (c) after harvest

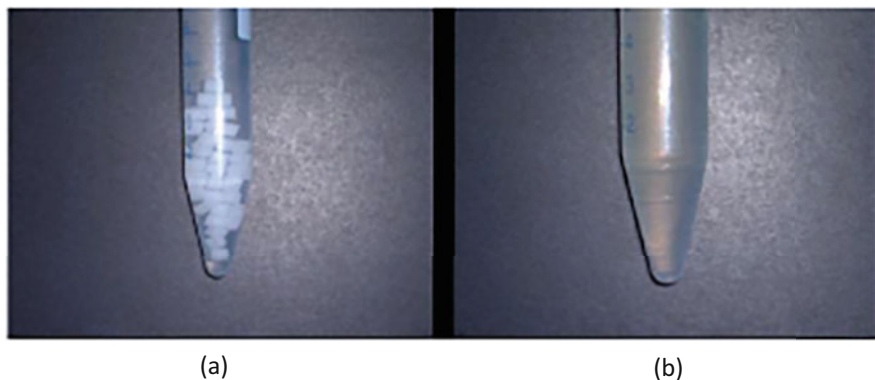


Fig. 24 Dissolution of PBS(MCC) pellets by hydrolysis accelerant. Hydrolysis accelerant: Enzyme provided by Novozymes A/S. (a) Original. (b) After 6.5 h at 50°C

3.9.3 Forest Protection Sheet

In the forestation business in the forest of Japan, wildlife nuisance that the planted young trees die caused by eating of tree bark by deer, etc. becomes sometimes problem. Conventionally, a cylindrical sheet made of PE or PVC has been used to protect the young tree. However, after they become unnecessary owing to the growth of the young tree, the sheet must be carried down the mountain and this needs much labor. So far as the biodegradable sheet is concerned, it is able to be embedded in the soil of the mountain, leading to reduction of the labor (Fig. 25). In other cases fumigation is sometimes adopted to prevent the outbreak of insect pests in felled trees and the felled trees are covered by sheets to prevent from the volatilization of the fumigant. The biodegradable sheet is occasionally used for this application. Applications for the reduction of the labor in the forest are expected to be developed more in the future.

3.10 Related Technological Developments and Topics

PBS exhibits mechanical properties analogous to polyethylene, and as for adaptive application, PBS is used for film applications analogous to polyethylene, such as agricultural films, plastic shopping bags, and garbage bags. But, when compared to LDPE, because PBS is lower in tear strength than LDPE, investigations regarding an improvement of tear strength have been made over the years. Because when used



Fig. 25 Protecting sheet for young trees

PBS alone, the tear strength is low, it is general to use it for a lot of applications after compounding with some kinds of polymers. As the polymer capable of enhancing the tear strength after compounding with PBS, there are known PBSA, PBAT, PCL, and the like. But, even if such a relatively flexible biodegradable resin is compounded, the tear strength does not reach that of LDPE. Then, various methods have been investigated. As for improvements regarding the tear strength so far, investigations such as kneading with starch, blending with polyethylene, addition of peroxide, adding of filler, and treatment with compatibilizer have been made. As an example, results of the investigation regarding an improvement of physical properties by blending with starch are given below.

3.10.1 PBS-Starch Compound

Because the starch is a natural material and is harmless and biodegradable, it is a material capable of adding to even agricultural mulching film and food packaging films. The starch is a natural polymer of a polysaccharide resulting from polymerization of α -glucose molecule by glycosidic linkage and is composed of amylose with a relatively low molecular weight resulting from polymerization of glucose by α -1,4 linkage and amylopectin with a high molecular weight having a structure in which besides the α -1,4 linkage, one unit per 25 units is linked with the α -1,6 linkage. The β -starch has a structure in a natural crystal state, and the α -starch has a structure in a state where the intermolecular hydrogen bonding is disjoined, and the molecular chains become free (Fig. 26).

As a hypothesis regarding the enhancement of the tear strength of PBS by the starch, it is conjectured that if the molecule of α -starch is able to take a structure in which it enters between the molecules of PBS, the hydroxy group of the starch and the ester group of polyester form water bond, and the tear strength is enhanced by intermolecular forces.

After mixing the starch with PBS and PBS/PBAT by an extruder, the film was molded through inflation molding, to measure the Elmendorf tear strength. Comparison between a polyethylene-made plastic shopping bag and a commercially available polyethylene-made garbage bag is shown (Fig. 27) [14]. The tear strength is a measured value in the MD (machine direction) direction.

According to a composition which is rich in PBAT, the tear strength was more enhanced than polyethylene. The tear strength was measured by varying the composition ratio in PBS and PBAT while fixing the addition amount of the starch to 30 parts (Fig. 28). When the ratio of PBAT is 50% or more, the enhancement of the tear strength is remarkable, but when the ratio of PBAT is lower than 50%, the tear strength tends to be lowered. Further, the relation with the tear strength was examined by varying the addition amount of the starch at a blending ratio of PBS and PBAT of 35/65 and 70/30, respectively. As a result, when the ratio of PBAT was 65%, the tear strength increased in accordance with the addition amount of the starch, whereas when the ratio of PBAT was 30%, the improving effect of the tear strength was not much revealed even through addition of the starch (Fig. 29). The

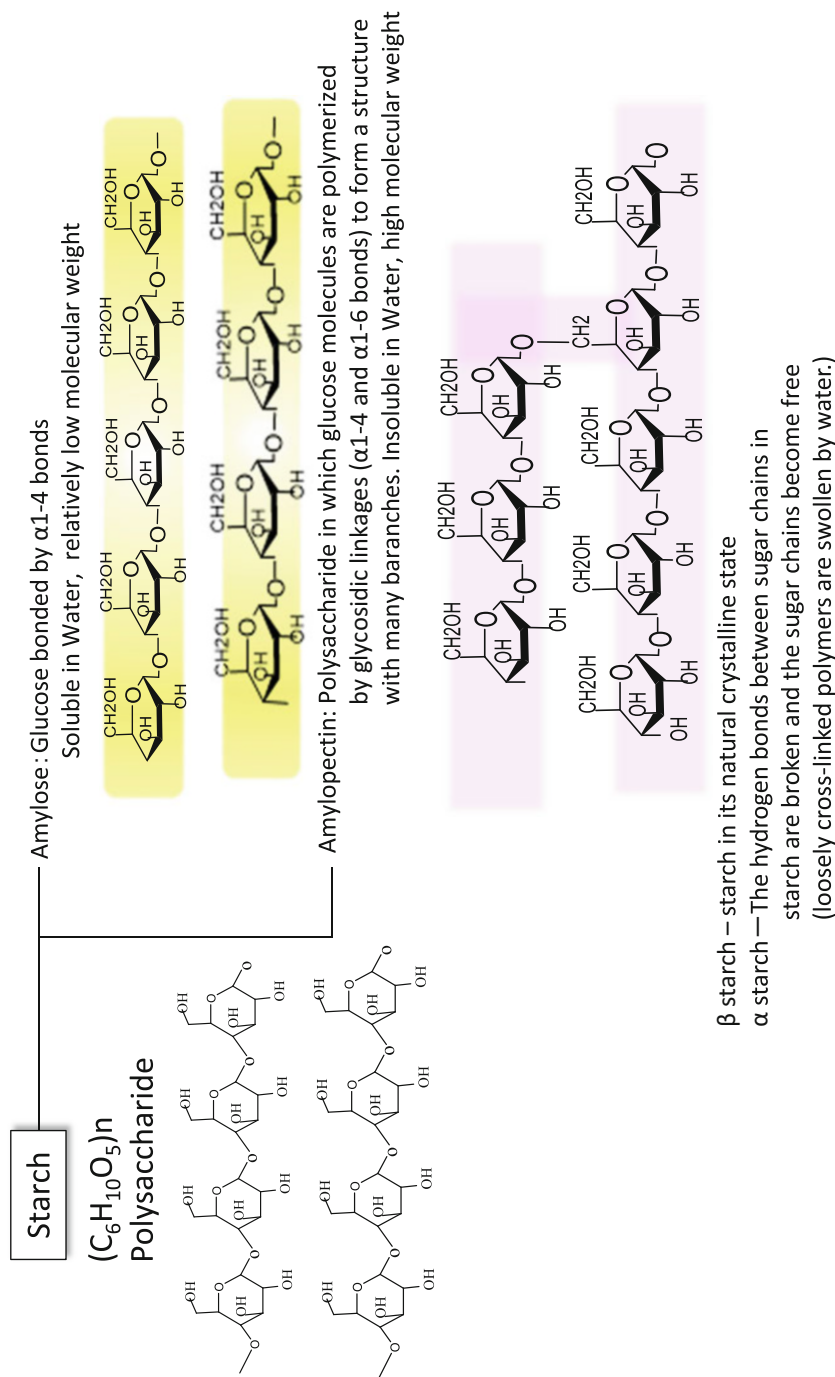


Fig. 26 Structure of starch

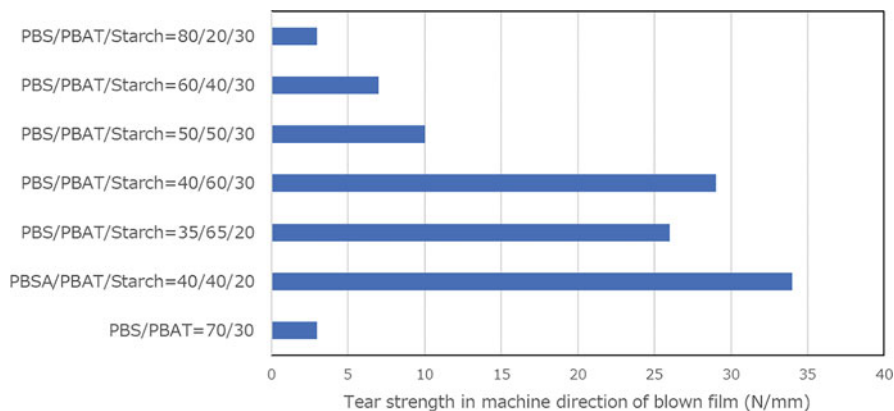


Fig. 27 Elmendorf tear strength of starch compounded PBS film

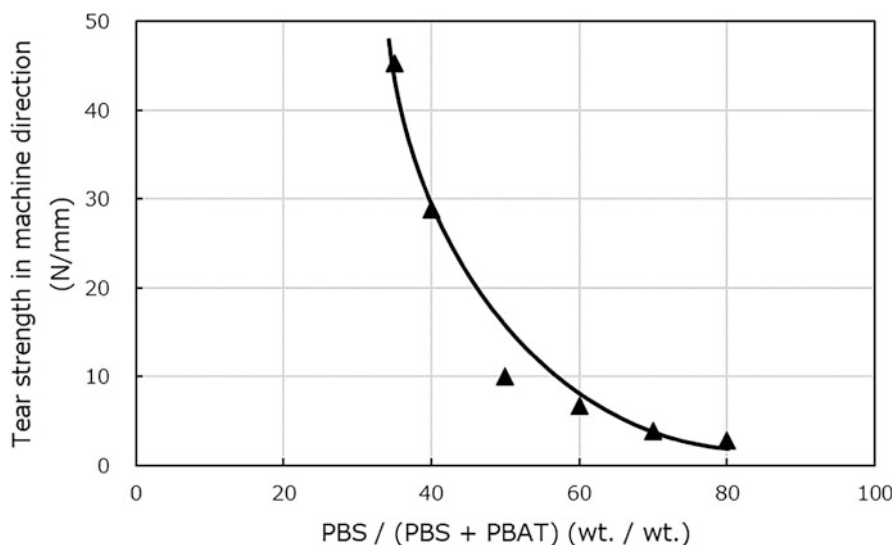


Fig. 28 Relationship between compounds ratio and tear strength of starch compounded film. Content of starch: 30 phr

state of the starch in the PBS/PBAT blend film was observed with an electron microscope. It has become clear that the starch particles exist in the portion of PBAT (Fig. 30).

The biodegradability in the soil of films having the starch mixed in each of PBS/PBAT and PBSA/PBAT was examined. Both exhibited good biodegradability. The PBSA/PBAT/starch blend film was slower in the biodegradability in the soil than PBSA (Fig. 31). Further, the behaviors the biodegradable films in the soil of the PBA/PBAT/starch and PBSA/PBAT/starch blend films are shown in Fig. 32.

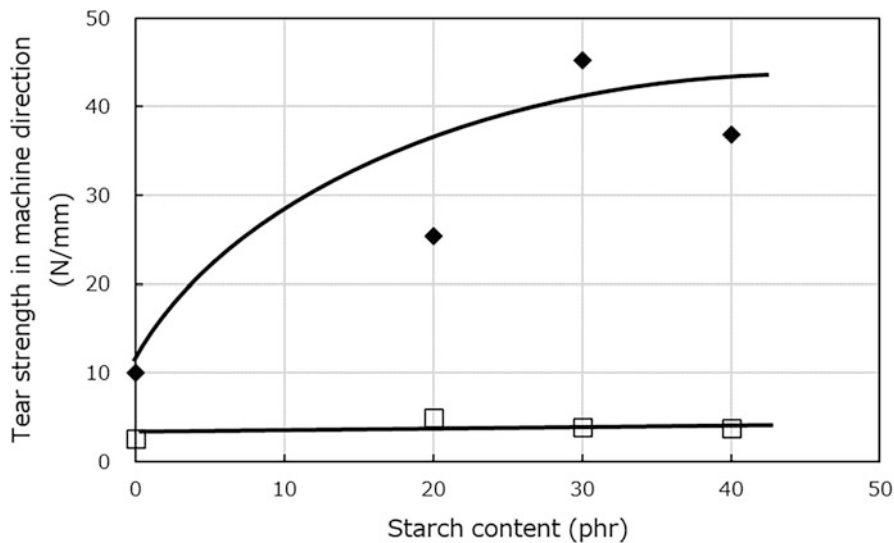
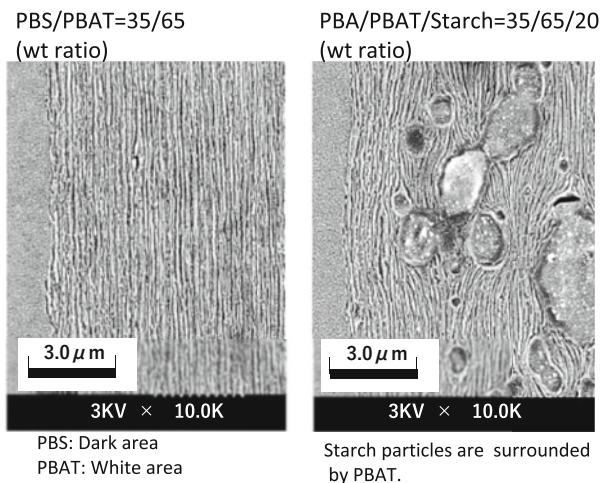


Fig. 29 Relationship between starch content and tear strength. Filled diamond: PBS/PBAT(=35/65) + starch. Open square: PBS/PBAT(=70/30) + starch

Fig. 30 Morphology of PBS/PBAT/starch film. PBS: Dark area. PBAT: White area



3.10.2 Enzyme Degradation of PBS

PBS is completely biodegraded with microorganisms in the natural world. However, because PBS has a high molecular weight, it is thought that PBS is first lowered in the molecular weight and oligomerized due to degradation of the ester linkage with an enzyme, and then degraded into carbon dioxide and water with microorganism.

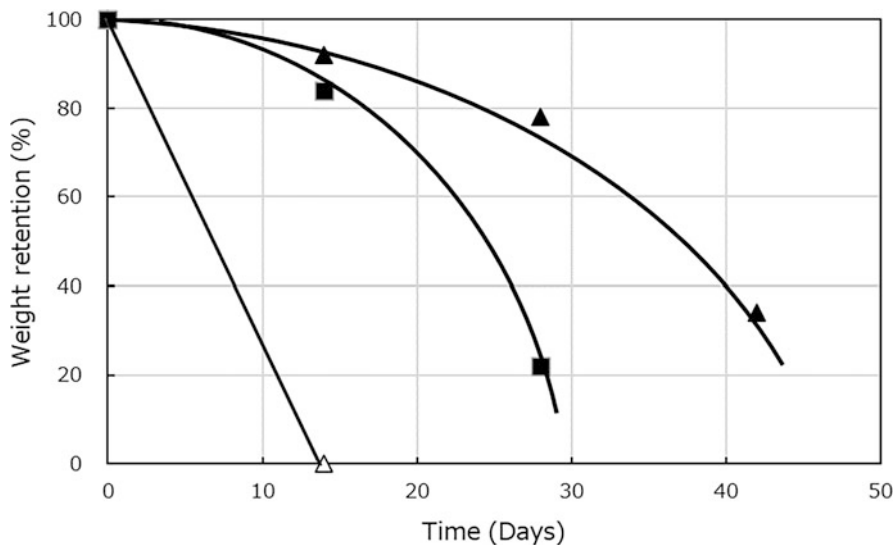


Fig. 31 Biodegradation test in soil at 50°C. Open triangle: PBSA. Filled triangle: PBSA/PBAT/starch = 50/50/15 (weight ratio). Filled square: PBS/PBAT/Starch = 35/65/20 (weight ratio)

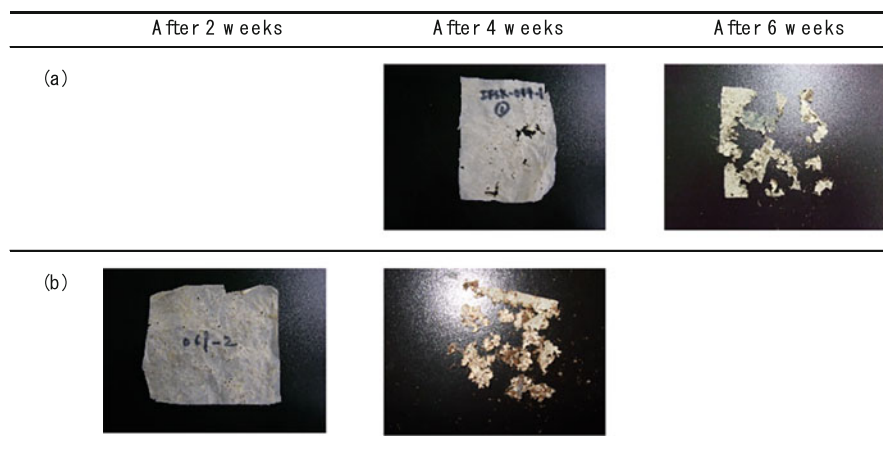


Fig. 32 Appearance changes of films through the biodegradation test in soil at 50°C. (a) PBSA/PBAT/starch = 50/50/15 (weight ratio). (b) PBS/PBAT/starch = 35/65/20 (weight ratio)

Mizuno et al. performed enzymatic degradation of PBS and PBSA to report that *Aspergillus niger*-derived lipase degrades PBSA [15]. Further, Maeda et al. report that *Aspergillus oryzae*-derived cutinase degrades PBS and PBSA [16].

Mitsubishi Chemical investigated promotion of the degradation with an enzyme in order to control the biodegradability of PBS. As for the PBS degrading enzyme, it was found that an enzyme that is a kind of *Humicola insolens* lipase produced from a

Table 14 Hydrolysis of PBS by lipase from *Humicola insolens* and other commercial lipases

| Enzyme | Degradation rate (%) of PBS film (30 μm thickness) | |
|--------------------------------------|--|------------|
| | 1 h (40°C) | 3 h (40°C) |
| Lipase from <i>Humicola insolens</i> | 54 | 100 |
| Lipases | | |
| <i>Aspergillus niger</i> | 0 | 0 |
| <i>Candida rugosa</i> | 0 | 0 |
| <i>Penicillium roqueforti</i> | 0 | 0 |
| <i>Burkholderia cepacia</i> | 0 | 0 |
| <i>Pseudomonas fluorescens</i> | 0 | 0 |
| <i>Achromobacter sp.</i> | 0 | 0 |
| <i>Candida cylindracea</i> | 0 | 0 |
| <i>Alcaligenes sp.</i> | 0 | 0 |

filamentous fungus *Aspergillus* strain exhibits very high biodegradability against PBS (Table 14) [17]. When PBS pellets were immersed in an enzyme aqueous solution at 50°C for 6.5 h, the pellets were completely degraded (Fig. 24) [18].

4 Future Outlook

At present, we face risks on a global scale, such as an increase of climate change and depletion of natural resources, uneven distribution of water resources, population increase and population ageing, and food and agricultural issue. Given this critical situation, as a chemical company, Mitsubishi Chemical believes that it is our mission to realize, through innovation, the efficient use of natural resources and energy, the utilization of renewable resources, and the reduction of environmental burden, thereby enhancing environmental and social sustainability.

Grappling with substitution of the raw material of plastic from petroleum as exhaustible resources with a biomass raw material as renewable resources realizes efficient use of resources and significantly contributes to ensuring sustainable production. Meanwhile, by giving biodegradability while making the most of characteristics of plastics, it becomes easy to achieve degradation in the environment, and it is also possible to contribute to reduction of environmental load. Though BioPBS™ and FORZEAS™ have both of these characteristic features, we expect that such materials will become a valuable solution in solving the issue on a global scale.

In the future, Mitsubishi Chemical will realize excellent materials close to the needs of clients through innovation and contribute to sustainable development of the global environment and society.

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