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A Study on Mechanical Properties of New Environmentally-friendly Construction Materials

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Doctoral Dissertation

A Study on Mechanical Properties of New Environmentally-friendly Construction Materials

> 環境に配慮した新しい建設材料の 力学特性に関する研究

> > January 2015

Graduate School of Agricultural Science, Kobe University

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The research reported in this dissertation was conducted at the Laboratory of Geotechnical and Environmental Engineering for Agricultural Land, Field of Agricultural Engineering and Socio-Economics, Graduate School of Agricultural Science, Kobe University, Japan.

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Notations

Chapter 2

q_u	:	Unconfined compression strength (kN/m ²)
k	:	Coefficient of permeability (cm/sec.)
С	:	Cohesion (kN/m ²)
ϕ	:	Friction angle (°)

Chapter 4

:	K ₂ SO ₄ -extractable total organic carbon (TOC) in soil
	(mg/kg)
	: Analysis value of K ₂ SO ₄ -extractable NPOC
	(non-volatility organic carbon) in soil (mg/L)
:	Analysis value of K ₂ SO ₄ -extractable NPOC in blank
	(mg/L)
:	Volume of 0.5 mol K ₂ SO ₄ -extract (ml)
:	Water in soil (ml)
:	Dry weight of extracted soil (g)
:	Carbon extracted by K_2SO_4 from fumigated soil (mg/kg)
:	Carbon extracted by K ₂ SO ₄ from non-fumigated soil
	(mg/kg)
:	Microbial biomass carbon (mg/kg)
:	0.45 (empirical conversion factor, Wu et al., 1990)

f_b	:	Flexural strength (N/mm ²)
р	:	Maximum load (N)
l	:	Support span (mm)
b	:	Width of specimen (mm)
h	:	Height of specimen (mm)
f_c	:	Compressive strength (N/mm ²)
Α	:	Loading area (mm ²)

Chapter 5

t	:	Time
α	:	Shape parameter
β	:	Scale parameter
Ν	:	Number of total sample
n	:	Order of data
MTTF	:	Mean time to failure
σ	:	Standard distribution
Г	:	Gamma function

CHAPTER 1

Chapter 1

Introduction

1.1 Environmental Issues

Since the industrial revolution, an age of abundance has come and accelerated. With rising population, the thoughts of mass production, mass consumption, and mass disposal have been established. On another front, it has had considerable effects on global-scale environmental destruction. There are many environmental issues, such as the pollution of atmosphere, water and soil, global warming, resource depletion and waste generation. We have traded beautiful nature for a life of abundance. At a certain point, it started to increase interest in environmental issues. It is said that this was the time when Rachel Carson established 'Silent Spring'.

•The history of life on earth has been a history of interaction between living things and their surroundings. ...Only within the moment of time represented by the present century has one species –man- acquired power to alter the nature of his world. (Rachel L. Carson, 2000a)

• To have risked so much in our efforts to mould nature to our satisfaction and yet to have failed in achieving our goal would indeed be the final irony. Yet this, it seems, is our situation. (Rachel L. Carson, 2000b)

We started to express our concern that our future life will not be in good shape if environmental destruction is continued. Therefore, consideration of environmental conservation such as recycling and green consumer and eco-friendly products were given rise.

1.2 Aim of This Study

The aim of this study is to create new environmentally-friendly construction materials.

The first study focused on the imminent environment. The theme is on the recycling of muddy soil deposited on small earth-fill dams. Many of small earth-fill dams are located in the Hyogo prefecture. Some problems are encountered when old small earth-fill dams are reconstructed. First, tons of muddy soil are deposited on the bottom of these dams, and it is necessary to remove them because muddy soil has bad smell and reduces the total volume of available water. Secondly, expensive soils for the impermeable core zone have to be prepared because of a lack of suitable soil materials at the dam sites. Hence, reuse of muddy soil for impermeable core zone of small earth-fill dams is proposed.

Another study focused on biodegradability. The theme is a new temporary material; biodegradable resin concrete. The manufacturing of biodegradable resin concrete was inspired by biodegradable plastic. Temporary materials like piles and sheet piles made of steel or concrete are left underground after construction if pulling them out has a negative effect on the surrounding soil. However, temporary structures may remain a continuous threat in terms of redevelopment and ground settlement due to the deterrent of construction and aging, respectively. Thus, biodegradable resin concrete that reverts to nature after a few years is considered beneficial. In this thesis, the rate of deterioration of biodegradable resin concrete is evaluated from some mechanical tests.

The consistent theme of these two studies is 'eco-friendly materials to promote a recycle-oriented society'.

1.3 Literature Review

In this section, the previous studies on environmental effort in agricultural engineering, small earth-fill dams and muddy soil, and biodegradable resin manufactures are presented.

1.3.1 Environmental Effort in Agricultural Engineering

Agriculture has strong links with the environment. In particular, the thought of sustainable agriculture is perceived as important in the last few decades. The development of agricultural engineering has led to increased crop harvest. On the other hand, the environmental load has been increased. Therefore, many efforts have been put into resolving many environmental issues in agricultural engineering.

Over the years, our laboratory has studied any environmental issues and how to solve them. For example, the methods of liquefied stabilized soil using sludge generated at a construction site (Kawabata et al., 2009), reuse of muddy soil deposited on bottom of ponds (Uchida et al., 2006) and slope reinforcement construction method using timber from bamboo forest thinning (Sawada et al., 2014) were proposed.

1.3.2 Studies on Small Earth-fill Dams and Muddy Soil

Many small earth-fill dams were constructed over a long period of history; it is said the oldest small earth-fill dams are Yosami-ike, Karisaka-ike and Kaeori-ike which were constructed in about 360 (Tani et al., 1998). Small earth-fill dams have played an essential role in agriculture as a valuable water source. However, these old earth-fill dams have been faced with disasters such as earthquakes and typhoons. Therefore, Hori et al. (1997 and 2002), Yamamoto et al. (1998), and Takagi et al. (2013) described and summarized the damage of small earth-fill dams. In these reports, the cause of the

collapse was clarified from field surveys and some soil tests.

Some reconstruction methods for many decrepit small earth-fill dams were devised (Tani, 2001 and 2002). Above all, many reconstruction methods using muddy soil have been reported (Fukushima et al. 2003, 2007 and 2009). Many researchers have studied muddy soil. Muddy soil has been mixied with other recycling materials, such as lime or gypsum agent (Shimokubo, 2007) and paper sludge ash (Mizuno, 2013).

The study on muddy soil in this thesis is different from previous studies. Muddy soil and old banking material (decomposed granite soil) are used. Both of them are generated at a construction site. Therefore, the improved soil made of muddy soil and old banking material is easily reused.

1.3.3 Studies on Biodegradable Resin Manufactures

Literature on biodegradable resin concrete has not been published because biodegradable resin concrete is a new product. Biodegradable resin was made in the 1980s (Japan BioPlastics Association, 2009a). The biodegradable resin itself has a short history. In recent years, it has been used in many fields, from medical science to agriculture. In the field of medical science, biodegradable resin is increasingly used as primarily needle and suture thread (Aoyagi et al., 2007). In the field of agriculture, it is used as mulching materials, seedling pots and nets for voluble stem (Japan BioPlastics Association, 2009b and Bernardo D. et al., 2000).

There has been interest in biodegradable resin and its mechanical properties have been studied (Kitamoto, 2009; Satoh et al., 2003). In the fields of engineering, the biodegradable resin is applied to some manufactures such as a bag for sandbag and a rope (Japan BioPlastics Association, 2009c and Nishimura et al., 2007). These manufactures are flexible materials. On the other hand, biodegradable resin concrete is a rigid material. Therefore, it is very important to understand its mechanical characteristics.

1.4 Overview of This Thesis

The overview of this thesis is shown in **Figure 1.1**. Chapter 1 provides a brief introduction, and the aim of this thesis. This chapter also provides an overview of environmental issues.

This thesis is composed of two parts. In Part 1, the reuse technique using muddy soil and other recycled materials is described. Part 1 is composed of Chapters 2 and 3. Chapter 2 explains the method of laboratory experiments and describes the results. In this chapter, the best fraction of improved soil is determined. Chapter 3 explains the method of field experiments and describes the results. This chapter suggests a possible beneficial effect of improved soil using muddy soil.

In Part 2, biodegradable resin concrete is described. Part 2 is composed of Chapter 4 and 5. Chapter 4 explains the experiments and describes the degradation characteristics and the degrading factors of biodegradable resin concrete. Additionally, the chapter describes practical applications of the biodegradable resin concrete. Chapter 5 explains a few model equations of deterioration prediction of biodegradable resin concrete.





Figure 1.1 Overview of this thesis

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Part 1

Improved Soil Using Muddy Soil

CHAPTER 2

The contents of this chapter are based on:

- Kawabata, T., Suzuki, M., Uchida, K. and Suzuki, T. (2011): Mechanical behavior of blended geo-materials for impermeable core zone of small earth-fill dams using recycle materials, 5th International Symposium on Deformation Characteristics of Geomaterials, pp.762-765
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Chapter 2

Laboratory Experiment of Improved Soil

2.1 Introduction

A small earth- fill dam; called Tame-ike in Japan, is defined as follows, it is an artificial pond with a the height under 15 m. Japan has approximately 210,000 small earth-fill dams and many of them are located in the Hyogo prefecture. The number exceeded 40,000 (Ministry of Agriculture, Forestry and Fisheries, 2014). Many of them were constructed over a long period of time, and have played an essential role in agriculture as a valuable water source; because steep and short Japanese rivers are not a stable source of water for rice farming.

Recently, the collapse of small earth-fill dams have become more frequent (**Photo 2.1**). More than 70% of small earth-fill dams were made before the Edo period, and have been faced with disasters such as earthquakes and typhoons. Prompt reconstructions of these aging small earth-fill dams are required in order to keep using them effectively.

The soil materials for impermeable core zone are required when a small earth-fill dam is reconstructed. Using the soil materials takes a high cost because the suitable soil materials cannot be collected near the dam sites. The soil materials need high quality in terms of the strength and impermeability. These earth-fill dams that need repair have a large deposits of muddy soil. Such muddy soil reduces the volume of water kept in the small earth-fill dams and leads to the deterioration of water. Though the muddy soil has to be removed, it is not easy to dispose of it because of high water content. In the past, many researchers have studied muddy soil to reuse (Fukushima et al., 2002, and Uchida et al., 2006 and 2010).

In this chapter, from laboratory experiments, the creation of the soil for impermeable core zone, using muddy soil deposited on the bottom of small earth-fill dams and some industrial wastes such as the old dam body's material, fly ash, crushed-stone powder, blast furnace slag and quicklime are proposed. The goal of this study is to solidify muddy soil without cement, and to create the soil for the impermeable core zone in the dam body mechanically stably, in an environmentally-friendly way and for a low price. The laboratory experiment methods are mainly the unconfined compression test and the permeability test.



Photo 2.1 Damaged small earth-fill dams

2.2 Materials

2.2.1 Muddy Soil

An individual small earth-fill dam has its own property of muddy soil. Muddy soil was dredged and some soil tests were conducted. The properties of the muddy soils are shown in **Table 2.1** and the grain size accumulation curve of Hazesara Pond is shown in **Figure 2.1**. In this laboratory experiment, the muddy soil from Kaisaka Pond (**Photo 2.2**) and Hazesara Pond (**Photo 2.3**) were mainly used, because at that time, the two small earth-fill dams were reconstructed. Muddy soil collected from Kaisaka Pond was used at a reduced water content of 150%. On the other hand, muddy soil of Hazesara Pond was used to make a homogeneous sample in the following process. First, the muddy soil (fresh sample from bottom of Hazesara Pond) was put in the drying furnace of 110°C and was made absolutely dry. Next, the dry muddy soil was crushed with the crushing machine into powder.

2.2.2 Decomposed Granite Soil

The decomposed granite soil is used in many of earth-fill dams' bank bodies because it is easy to compact (HAM et al., 2002). Two types of decomposed granite soil were used in this study. One was obtained from Ono city in Hyogo prefecture and another was obtained from Awaji Island. The decomposed granite soil from Ono city had enough fine fraction. It is considered that the fine fraction has flushed out from old bank bodies which need to reconstruct. Therefore, the size distribution of the decomposed granite soil from Ono city was regulated not less than 75 μ m and not more than 2 mm. The properties and the grain size accumulation curve of two decomposed granite soil is shown in **Table 2.2** and **Figure 2.1**.
Table 2.1Properties of muddy soils							
	Kaisaka Pond	Hazesara Pond	Nishiohtani Pond	Nanbu Tameike			
Density of soil particles (g/cm^3)	2.560	2.650	2.575	2.580			
Natural water content(%)	366.5	149.8	-	145.0			
Liquid limit (%)	165.6	148.4	81.50	142.0			
Plastic limit (%)	116.2	58.10	39.30	46.80			
Plasticity index	49.39	86.54	-	95.20			
Lignition loss (%)	16.01	11.79	-	10.40			



Figure 2.1 Accumulation curve of muddy soil and decomposed granite soil



Photo 2.2 Muddy soil from Kaisaka pond

Photo 2.3 Muddy soil from Hazesara pond

•1

Table 2.2 Properties of	of decomposed granite soils							
	Ono city	Awaji Island						
Density of soil particles (g/cm ³)	2.670	2.690						
Maximum dry density (g/cm ³)	1.760	1.780						
Optimum moisture content(%)	15.00	15.08						

2.2.3 Fly Ash

Fly ash, shown in **Photo 2.4**, is recovered as a by-product when pulverized coal is burnt in a coal-fired station. The particle size is $0.5 \sim 100 \,\mu\text{m}$. The fly ash used in this study was discharged from Hekinan Thermal Power Station, Chubu Electric Power Co., Inc. The amount of emergence is increasing each year, so it is counted on its efficient use (Shibata, 2008). In the chemical composition, the majority is silicon dioxide and oxidized aluminum; therefore it is a solidification supplementary agent that can be expected in terms of the long-term strength appearance by the latent hydraulic property. Additionally, it has a large surface area and a good adhesive property, because the bulk specific gravity is small and the particle is porous.

2.2.4 Quicklime

The principal ingredient of the quicklime as shown in **Photo 2.5** is a calcium oxide. When the quicklime changes into hydrated lime in soil, the volume of water is absorbed, and it becomes solidified under a chemical reaction as time passes. Furthermore, cohesive soil, organic soil, and sludge can be solidified, if the pozzolana material is efficiently mixed like a slag (Japan Lime Association, 2014). Quicklime used in this study is manufactured by Uedalime manufacturing Co., Ltd.



Photo 2.4 Fly Ash

Photo 2.5 Quicklime

2.2.5 Granulated Blast Furnace Slag

Granulated blast furnace slag (**Photo 2.6**) is a by-product obtained in the manufacturing of pig iron in the blast furnace and is formed by the combination of earthy constituents of iron ore with limestone flux. The amount of the by-product is increasing each year, suggesting efficiency in reducing waste (Nippon Slag Association, 2014). The grain size accumulation curve is shown in **Figure 2.2**. The granulated blast furnace slag was used by focusing on the latent hydraulicity, which can improve long-term strength (Kikuchi et al., 2006). **Table 2.3** shows the composition of the slag. The granulated blast furnace slag used in this study was obtained from Kobe Steel, Ltd.

2.2.6 Crushed-stone Powder

Crushed-stone powder (**Photo 2.7**) is also a by-product obtained in the manufacturing. Long-term strength can be expected by using crushed-stone powder with decomposed granite soil (Nishida et al., 1991). The crushed-stone powder used in this study is manufactured by OSAKA SAISEKI Co., Ltd. Crushed-stone powder was sieved to adjust its particle size to under 2 mm. The grain size accumulation curve is shown in **Figure 2.2**.



Photo 2.6 Granulated blast furnace slag

Photo 2.7 Crushed-stone powder



Figure 2.2 Accumulation curve of granulated blast furnace slag and crushed- stone powder

Table 2.3 Co	mpositi	on of g	ranulate	d blast f	urnace s	lag (Ni	ppon Sla	ag Assoc	iation, 2	.009)
	CaO	SiO_2	T-Fe	MgO	Al_2O_3	SiO_2	P_2O_5	MnO		
	41.7	33.8	0.4	7.4	13.4	0.8	< 0.1	0.3		

2.3 Test Methods

2.3.1 Unconfined Compression Test

The unconfined compression test was conducted to clarify the strength of improved soil. The important point is that which compounding ratio has the most effect of improvement, and what the mechanism of strength increase is. The criteria for unconfined compression strength is $q_u = 65 \text{ kN/m}^2$ (early-age strength) and $q_u = 143 \text{ kN/m}^2$ (seven day strength) (Fukushima et al., 2002). The early-age strength ($q_u = 65 \text{ kN/m}^2$) is known as 'trafficability', which is the ability of a terrain that vehicles and troops are able to pass.

The specimen diameter is 50 mm and the height is 100 mm. The equipment and the specimen are shown in **Photo 2.8**. The specimen was compacted in four layers into a cylindrical mold. It was compacted nine times per layer with the 1.5 kg rammer. The

method of preparing for specimens was according to JIS A 1210. Furthermore, three specimens were made per mixture pattern.

Some specimens which yield to long-term strength mixing with latent hydraulicity materials such as fly ash, crushed-stone powder and granulated blast furnace slag were cured in 3, 7 and 28 days. The curing methods are two pattern, atmospheric curing and pressure curing. Design requirements for the core material included not only low permeability and high strength, but also resistance to shrinkage on drying. However, improved soil is hard to dry because it is constructed gradually. In this laboratory test, the specimens were kept moist. The loading speed was 1% per minute.

2.3.2 Permeability Test

Permeability of soil is very important to estimate stability of the small earth-fill dams. The coefficient of permeability criterion for impermeable core zone of small earth-fill dams is known to under $k = 5.0 \times 10^{-6}$ cm/sec. (The Japanese Society of Irrigation, Drainage and Rural Engineering, 2000). The method of the permeability test is prescribed by JIS A 1218. The permeability test was examined for 4 cases. The specimen had a cubical size of 156 mm. The equipment and the specimen are shown in **Photo 2.9**.



Photo 2.8 Unconfined compression test equipment and a specimen



Photo 2.9 Permeability test equipment and a specimen

2.3.3 Scanning Electron Microscope (SEM) Observation

Chemical analyses of specimens of some improved soils were performed by Scanning Electron Microscope (SEM). SEM observation is able to explain the reason of strength increase.

The famous chemical reaction of fly ash and granulated blast furnace slag is pozzolanic activity. A matter of common knowledge is that ettringite $(3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O)$, calcium silicate hydrate $(nCaO \cdot SiO_2 \cdot mH_2O)$ and calcium aluminate hydrate $(3CaO \cdot Al_2O_3 \cdot 6H_2O)$ are formed by pozzolanic activity (Ogawa et al., 1980). Acicular microcrystals and some colloids are observed by SEM if pozzolanic activity occurs.

2.4 Compounding Ratio

The compounding ratio of this test is shown in Table 2.4.

In Case 1, muddy soil from Kaisaka pond and decomposed granite soil from Ono city are the main materials. The water content of all specimens except A is 25%. First, in order to clarify the base material, the decomposed granite soil from Ono city and the crushed-stone were compared (from Case 1-A to 1-E). Second, Case 1-F, 1-G and 1-H shed light on the best compounding ratio. Finally, the long-term strength of the fly ash is clear in Case 1-I and 1-J.

In Case 2, the muddy soil from Hazesara pond and the decomposed granite soil from Awaji Island are the main materials. The water content of the specimens is adjusted to the optimum moisture content (OMC). The best compounding ratio between the muddy soil from Hazesara pond and the decomposed granite soil from Awaji Island is clarified from Case 2-K to Case 2-N. The fly ash and quicklime are kinds of admixtures which are mixed 3, 5 and 10%. In Case 2, the fly ash and the quicklime were mixed with outer percentage. In Case 2-P to Case 2-S, the granulated blast furnace slag was used instead of the decomposed granite soil.

	Case 1									
	А	В	С	D) E	F	G	Η	Ι	J
muddy soil from Kaisaka pond		43	33	3 43	3 33	17	17	17	26	26
decomposed granite soil from Ono city	100	57	67	7		56	63	67	46	52
fly ash									5	5
crushed-stone powder				51	7 67	28	21	17	23	17
water content (%)	OMC					25				
					Case	2				
	Κ	L	Μ	Ν	0	Р	Q	R	1	S
muddy soil from Hazesara pond		50	33	20	20	50	33	30	3	30
decomposed granite soil from Awaji Island	100	50	67	80	80					
fly ash					3~10%					
quicklime					3~10%				5~	10%
granulated blast furnace slag						50	67	70	7	0'0
water content (%)					OMO	2				

Table 2.4 Compounding ratio

2.5 Results and Discussion

2.5.1 Comparison of Decomposed Granite Soil from Ono City with Crushed-stone Powder (from Case 1-A to Case 1-E)

Figure 2.3 shows the unconfined compressive strength of the only decomposed granite soil from Ono city. The unconfined compressive strength was under 65 kPa. This decomposed granite soil is a substandard material. Note that the size distribution of this decomposed granite soil from Ono city was regulated in the range of 75 μ m to 2 mm, to simulate the old banking material.

Figure 2.4 shows the comparison between the decomposed granite soil from Ono city and the crushed-stone powder. The grain size distribution of this crushed-stone powder is similar to the grain size distribution of the decomposed granite soil. Therefore, crushed-stone powder could be used as the base material of small earth-fill dams instead of decomposed granite soil. However, contrary to our expectation, it was obvious that the crushed-stone powder did not show enough unconfined compressive strength. This is because the crushed-stone powder was not integrated into muddy soil. The ease of the blending stems largely from the shape of the particle. The form of a particle of decomposed granite soil is relatively circular. On the other hand, the crushed-stone powder is an angular particle. The difference of strength between the decomposed granite soil and the crushed-stone powder is attributed to the difference of form of these particles.

The proportion of the muddy soil and the decomposed granite soil is an important. The unconfined compressive strength was below 65 kPa considered to be safety criteria for small earth-fill dams if the proportion of the muddy soil is over 40%.



Figure 2.4 Unconfined compressive strength of from Case 1-B to Case 1-E

2.5.2 The best Compounding Ratio of Muddy Soil from Kaisaka Pond, Decomposed Granite Soil from Ono City and Crushed-stone Powder (Case 1-F, Case 1-G and Case 1-H)

Figure 2.5 shows the unconfined compressive strengths of the different rate of the muddy soil from Kaisaka pond, the decomposed granite soil from Ono city and the crushed-stone powder. Case 1-H did not meet criteria. It means that the high unconfined compressive strength is not shown if a high percentage of the decomposed granite soil is blended, suggesting the cause of the water content and the grain size distribution. Samples in Case 1-H contain about 70% decomposed granite soil. The optimum moisture content of this decomposed granite soil is about 15%. However, the water content of these three improved soils was 25%. The water content of Case 1-H is separated from the optimum moisture content although the most part of blend material was the decomposed granite soil. As additional experiment, specimens with 20% of water content were made and unconfined compression tests were conducted. All specimens of 20% water content met criteria.

As discussed previously, the water content and the grain size distribution are important in expressing the adequate unconfined compressive strength.



Figure 2.5 Unconfined compressive strengths of Case 1-F, Case 1-G and Case 1-H

2.5.3 Long Term Strength by Fly Ash (Case 1-I and Case 1-J)

Figures 2.6 and **2.7** show the long term strength by the fly ash. In Case 1-I, the longer period specimens were cured, the more unconfined compressive strength increases. However, in Case 1-J, little strength increase was shown. It is said that pozzolanic activity of fly ash is accelerated, after material age is over 91 days (Yamamoto et al., 2007).





2.5.4 Comparison of Decomposed Granite Soil from Awaji Island with Granulated Blast Furnace Slag (from Case 2-K to Case 2-N and from Case 2-P to Case 2-R)

Figure 2.8 shows the unconfined compressive strengths of the different rate of the muddy soil from Hazesara pond and the decomposed granite soil from Awaji Island (from Case 2-K to 2-N). From the figure, it is obvious that the decomposed granite soil increases its unconfined compressive strength as the ratio of muddy soil increases. A small earth fill-dam's impermeability core zone needs to have a compressive strength of 65 kN/m² at the minimum (Fukushima et al., 2002).

The decomposed granite soil used in this experiment has a large fine fraction, so it conforms to standards of safety easily. Furthermore, the muddy soil used in Case 2 was premeasured to make a homogeneous sample. It is thought that this process led to higher strength than Case 1. It is important that each material should be blend homogeneously without aggregating soil particles.

Figure 2.9 shows the unconfined compressive strengths of the different rate of the muddy soil from Hazesara pond and the granulated blast furnace slag (from Case 2-K to 2-N). From the figure, it is obvious that the unconfined compressive strength grows as the ratio of muddy soil increases. From **Figure 2.9**, it is clear that the granulated blast furnace slag can meet criteria by the addition of approximately 30% muddy soil.



Figure 2.8 Unconfined compressive strengths of from Case 2-K to Case 2-N



Figure 2.9 Unconfined compressive strengths (from Case 2-P to Case 2-R)

Figure 2.10 shows the comparison of the decomposed granite soil from Awaji Island to the granulated blast furnace slag. The unconfined compressive strength of Case 2-L and Case 2-P, which have the same rate of muddy soil (contained 50% muddy soil), were nearly identical to each other. However, the unconfined compressive strengths of Case 2-M and Case 2-Q, which have the same rate of muddy soil (contained 33% muddy soil), made a large difference. The cause was considered to be the difference of the cohesion and the internal friction angle of the decomposed granite soil and the granulated blast furnace slag. Then, the direct shear test was conducted and the cohesion and internal friction angle were obtained. Additionally, water absorption test (JIS A 1109) was conducted. The results are shown in **Table 2.5**. The difference of unconfined compressive strength is attributed to cohesion and water absorption.



from Awaji Island and granulated blast furnace slag

	$c (kN/m^2)$	\$\$\phi\$\$ (°)	water absorption (%)
decomposed granite soil from Awaji Island	21.52	37.6	3.84
granulated blast furnace slag	0.8968	42.3	0.54

Table 2.5 Cohesion, internal friction angle and water absorption

2.5.5 Long Term Strength by Fly Ash and Quicklime (Case 2-O and Case 2-S)

In a practical situation, decomposed granite soil, which is collected from the dam site, might have less fine fractions and higher moisture content. Therefore, admixtures such as quicklime and fly ash will be required.

The impact of additions of quicklime and fly ash on the strength development of specimens made from decomposed granite soil and muddy soil are shown in **Figure 2.11**. The data presented in **Figure 2.11** indicates that the specimen to which 10% quicklime was added without curing was stronger than the specimens with the addition of 5% and 3% quicklime. It is inferred that this strength increase originated from the aggregate reaction with quicklime. Japan Lime Association (2010) shows that the aggregation action with quicklime is a kind of stable treatment; it is an action to which the soil particle was electrically bonded by the ion exchange reaction between the calcium ion of the quicklime and the soil. Specimens cured over a long period hardened depending on the amount of fly ash. However, after three days of curing under atmospheric pressure the specimen which was added to 5% quicklime + 3% fly ash, 10% quicklime + 3% fly ash and 10% quicklime + 5% fly ash was of lesser intensity than 0 day. It suggested that cracks by the cubical expansion due to the hydration of the quicklime in the specimen as



Figure 2.11 Long term strength by fly ash and quicklime (Case 2-O)

a possible cause of intensity decrease. Therefore, the long-term strength can be expected if quicklime is added to the same level as fly ash or a less amount.

The effect of improvement by the latent hydraulic property of the granulated blast furnace slag was examined. Besides, the difference depending on additive amount of quicklime was examined. The specimens blending 70% granulated blast furnace slag and 30% muddy soil were used in order to be easy to understand the effect of the improvement.

The data presented in **Figure 2.12** indicates that the addition of quicklime to granulated blast furnace slag is able to improve the strength development. The early strength increase is inferred to originate from the increase of fine fraction by quicklime. The unconfined compressive strength becomes about twice as much after 28 days. Thus, it can be said that quicklime is a good admixture for granulated blast furnace slag.

Moreover, without quicklime specimens (Case 2-R) were cured in order to reveal latent hydraulicity of granulated blast furnace slag by itself. As a result, the unconfined compressive strength was increased without quicklime although the increase of strength was less than other specimens with quicklime.



Figure 2.12 Long term strength of the blast furnace slag by quicklime (Case 2-R and Case 2-S)

2.5.6 Unconfined Compression Test after Pressure Curing

From the results as referred to section 2.5.5, the specimen mixed with 5% quicklime + 3% fly ash, 10% quicklime + 3% fly ash and 10% quicklime + 5% fly ash decreased strength greater than any other specimens (**Figure 2.11**). Consequently, the curing method was changed, and it was cured under confined pressure into the triaxial cell for 7days; after that, unconfined compression tests were conducted. The results are shown in **Figure 2.13**. The specimens were scarcely affected by cubical expansion and did not crack. The strength of the specimens which were cured by 50 kPa was higher than any other specimens.

2.5.7 Permeability Test

Figure 2.14 shows the permeability test results. It is obvious that the specimens of Case 2-N did not meet the safety standard value for the coefficient of permeability, although it met the criteria for unconfined compression strength.

The improved soil can be used after admixtures, quicklime and fly ash were added (Case 2-S). It suggested that the void might be filled with quicklime and fly ash. In fact, Case 2-P satisfied the safety standard value without admixtures. Consequently, if enough soil materials such as muddy soil and decomposed granite soil could be obtained, an earth-fill dam might be reconstructed at a lower cost because admixtures are not needed.



Figure 2.13 Unconfined compressive strength after pressure curing



Figure 2.14 Permeability test result

2.5.8 Scanning Electron Microscope (SEM) Observation

The observation by a scanning electron microscope was contracted to Research Equipment Center of Hamamatsu University School of Medicine. **Photo 2.10** shows the scanning electron micrograph of the 90 day cured specimen. The needle-like ettringite that was made from fly ash was not observed. However, the smoke-like structure was generated by fly ash and quicklime. It is said that this smoke-like structure is a kind of calcium silicate hydrate ($nCaO \cdot SiO_2 \cdot mH_2O$), and microstructure of specimens become densification by calcium silicate hydrate (Iwahara et al., 2008).

Figure 2.15 and **Photo 2.11** show X-ray analysis and scanning electron micrograph of the 90 days cured specimen. According to X-ray analysis, it is suggested that gel hydrate containing a high proportion of Al and Ca mineral from quicklime was increased.



Photo 2.10 Scanning electron micrograph of the 90 days cured specimen



Figure 2.15 X-ray analysis



Photo 2.11 Scanning electron micrograph of the 90 day cured specimen

2.6 Conclusions

In this chapter, from laboratory experiments, the creation of the soil for impermeable core zone of small earth-fill dams was proposed, using the muddy soil deposited on the bottom of a pond and other recycled materials, such as the granulated blast furnace slag, the old banking material of fine-grain fraction lack, the crushed-stone powder, the fly ash and the quicklime, which are mechanically stable. From the result of experimented investigation, the following conclusions were obtained.

- The suitable material to mix with the muddy soil is not the crashed stone powder, but the decomposed granite soil. The proportion of the muddy soil and the decomposed granite soil is important. It is necessary that the improved soil be blended after considering the water content and grain size distribution of the muddy soil and the decomposed granite soil.
- 2. The strength of the long-term strength can be anticipated if the quicklime and the fly ash are added. The strength of the quicklime should not exceed that of the fly ash, because the specimens would develop micro cracks and decrease the strength with curing under atmospheric pressure. However, from the results of pressure curing, the ratio of admixture was regarded as unimportant. It is better that the confined pressure of curing is 50 kPa.
- 3. The quicklime and the fly ash played a role by reducing the coefficient of permeability. It is clarified from the observation by a scanning electron microscope that long-term strength and permeability reduction stem largely from densification by calcium silicate hydrate.
- 4. The granulated blast furnace slag can be used instead of the decomposed granite soil. However, there is a large difference of cohesion and water absorption between the decomposed granite soil and the granulated blast furnace slag. Therefore, it is necessary to attend to the mixture proportion of the muddy soil.
- 5. The granulated blast furnace slag has latent hydraulicity on its own itself. Additional strength increase is expected if the quicklime is added.

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CHAPTER 3

The contents of this chapter are based on:

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Chapter 3

Field Experiment of Improved Soil

3.1 Introduction

Following the laboratory experiment results, a field experiment was conducted. In the field experiment, a large soil-recycling machine; SR-G2000 Track Mounted Soil Recycler shown in **Photo 3.1** was used to blend materials. This machine has been used for road embankment material and back-filling material (Kyusyu Branch of the Japanese Geotechnical Society, 2003). In this field experiment, this large soil mixing machine used to achieve homogeneous mixing was tested for practical applicability since it had not been used before during restoration work of small earth-fill dams under the use of the improved soil for the impermeable core zone. This field experiment was carried out at Tatsu pond in Kasai city, Hyogo prefecture.

3.2 Materials

The materials employed in this field experiment, which were the muddy soil (**Photo 3.2**), the old banking material; the decomposed granite soil (**Photo 3.3**) and the

cementitious solidification material for soft-ground. Though the quicklime and the fly ash should have been the same as in the laboratory experiment, it was difficult to transport the quicklime and the fly ash and the cementitious solidification material was used. The properties of the muddy soil are shown in **Table 3.1**. The properties and the grain size accumulation curve of old banking material is shown in **Table 3.1** and **Figure 3.1**.



Photo 3.1 SR-G2000 Track Mounted Soil Recycler



Photo 3.2 Muddy soil from Tatsu pond



Photo 3.3 Old banking material

Table 3.1Properties of muddy soil and old banking material



Figure 3.1 Accumulation curve of old banking material (decomposed granite soil) from Tatsu pond

3.3 SR-G2000 Track Mounted Soil Recycler

SR-G2000 Track Mounted Soil Recycler is a product of Hitachi Construction Machinery. The machine is used to stabilize soft soil and sediment such as muddy soil (Hitachi Construction Machinery GLOBAL, 2014). Figure 3.2 shows dimensional drawing of SR-G2000. The internal blender of this machine is the biaxial paddle method as shown in Photo 3.4. Therefore, various soil will be blended homogeneously.







2 軸パドルミキサ方式を継承

混合機には「2 軸バドルミキサ」方式を採用し、さまざまな性状の原料土に対応します。
Photo 3.4 Biaxial paddle method (Hitachi Construction Machinery, 2014)

3.4 Test Methods

First, the muddy soil and the old banking material were mixed without the cementitious solidification material using SR-G2000 Track Mounted Soil Recycler. The volume proportion of the muddy soil were identical to that of the old banking material. The numbers of times to mix materials were from one time to ten times. The improved soil samples were sampled each time. Wet density and water content of each soil samples were measured.

Next, the muddy soil, the old banking material and the cementitious solidification material were mixed using the machine. The volume proportion of the muddy soil and the old banking material is equal and the cementitious solidification material was added 50, 100, 150 kg to 1 m³ of soil. The numbers of times the materials were mixed were one time, three times and five times. The improved soil samples were sampled and made each time. The specimen diameter is 50 mm and the height is 100 mm. After curing for 28 days, the unconfined compression test was conducted. The loading speed was 1% per minute.

3.5 Results and Discussion

3.5.1 Unconfined Compression Test Result

Figure 3.3 shows the relationships between the amount of stabilizing cement and unconfined compressive strength of the improved soil for the different number of mixing times by a large soil mixing machine.

The mixture proportion was 50% of muddy soil and 50% of decomposed granite soil because a lot of muddy soil could be obtained from Tatsu pond. From the results shown in **Figure 3.3**, it was found that the more times the improved soil was blended, the more unconfined compressive strength was increased. The average value of the compressive strength showed 37.2 kN/m² when the improved soil was blended with stabilizing cement once. When blending three times, the average value of the compressive



Figure 3.3 Unconfined compression test associated with mixing time

strength showed 73.7 kN/m². When blending five times, the average value of the compressive strength showed 75.2 kN/m². The same reaction occurred when 100 or 150 kg/m³ of stabilizing cement was blended. This point indicates that the stabilizing cement was homogeneously mixed with the increase of mixing time.

Figure 3.4 and **Figure 3.5** show the variations of water content and wet density as a function of the number of times mixed, respectively. The degree of variation of water content decreases as the number of times mixed increases. Furthermore, the values of wet density tends to become higher with the increase of mixing time.



Figure 3.4 Relationships between water content and the number of times mixed



Figure 3.5 Relationships between wet density and the number of times mixed

3.6 Conclusions

In this chapter, the following findings were obtained through the field experiment associated with the use of muddy soil.

- In the case of a large soil mixing machine used in a construction field, improved soil should be scrambled up in several times. The number of times mixed is desirably three times at least.
- 2. The more times the improved soil was blended, the more the unconfined compressive strength increased, and the more homogeneous the improved soil became.
- 3. From the measured results, plausible values of the water content and the wet density may be achieved at least three times that of the mixture.

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Part 2

Biodegradable Resin Concrete

CHAPTER 4
The contents of this chapter are based on:

- Suzuki, M., Kubo, K., Suzuki, T. and Kawabata, T. (2014): Mechanical Approach to Eco-friendly Biodegradable Resin Concrete, *Concrete Research and Technology*, Vol.25, pp.119-124 (in Japanese)
- Suzuki, M., Kubo, K., Sawada, Y. and Kawabata, T. (2014): Mechanical Characteristics of Environmentally-friendly Biodegradable Resin Concrete, Concrete Innovation Conference 2014, USB date No.17, pp.1-7
- Suzuki, M., Kubo, K., Suzuki, T., Sawada, Y. and Kawabata, T. (2014): Effect of Different Exposure Condition on Degradation Characteristics of Biodegradable Resin Mortar, IRRIGATION, DRAINAGE AND RURAL ENGINEERING JOURNAL (submitted)
- Suzuki, M., Kubo, K. and Kawabata, T. (2014): Effect of Different Type of Resin on Degradation Characteristics of Biodegradable Resin Concrete, *Concrete Research* and Technology, (in Japanese) (plan on submitting)
- Suzuki, M., Kubo, K. and Kawabata, T. (2014): Apply of Biodegradable Resin Concrete to Propulsion Pipes, IRRIGATION, DRAINAGE AND RURAL ENGINEERING JOURNAL (Technical Notes) (submitted)

Chapter 4

Biodegradable Resin Concrete Experiment and Application

4.1 Introduction

Generally, temporary materials such as a pile and a sheet pile made of steel or concrete are left in the ground after construction if they have negative effects on the surrounding soil when pulled out. However, if commonly-used temporary materials have been left in the site, various problems for redevelopments will be generated in the future such as; (1) a decline in land worth, (2) industrial waste generation and (3) ground settlement. From a social background, biodegradable resin concrete is proposed to be used for temporary materials. Biodegradable resin concrete is inspired by biodegradable plastics. In recent years, there has been an interest in biodegradable plastics, and their mechanical properties have been studied (Kitamoto, 2009; Satoh et al., 2003). Owing to its outstanding environmental load reduced properties, biodegradable plastics have been used in many fields, from medical science to agriculture. On the other hand, biodegradable resin concrete is a new product. To the best of our knowledge, no studies relevant to biodegradable resin concrete have been reported. Therefore, it is very important to understand their mechanical characteristics.

In this study, some mechanical experiments on biodegradable resin concrete were

conducted to clarify the deterioration mechanism and for deterioration prediction.

The final goal of this study is to achieve the practical use of biodegradable resin concrete, such as a pile, a sheet pile, a temporary road, a slope face stabilizer and a pipeline. At the end of this chapter, some mechanical test results of biodegradable resin concrete pipe were describe as step toward the practical use. Biodegradable resin concrete pipes are expected to contribute to trenchless technology. It may be possible to remove an existing pipe with the jacking method, using a biodegradable resin concrete pipe.

4.2 Materials

4.2.1 Biodegradable Resin Concrete

Commonly-used resin concrete is combined using materials made from synthetic resin and aggregates instead of cement and aggregates. Traditional resin concrete mainly uses unsaturated polyester resin and epoxy resin (Takemura et al., 1998). On the other hand, the biodegradable resin concrete in this study uses biodegradable resin as a bond material. In ordinary circumstances, biodegradable resin is applied to light and soft products such as plastics, films and sheets. Therefore, this study provides new initiative because biodegradable resin is applied to hard products such as concrete.

The biodegradable resin concrete is made of aggregates of sand or gravel and biodegradable resin. Two kinds of biodegradable resin were used. One is BionolleTM#3001 (PBSA) and another is TERRAMAC TE-7300 (PLA). BionolleTM#3001, which was developed by SHOWA DENKO K.K, Japan. BionolleTM#3000 series is also known as polybutylene succinate adipate (PBSA). PBSA is substituted 20% succinic acid of PBS with adipic acid. PBS is copolymer of 1.4 butanediol and succinic acid (Asumi Lab. Japan, 2014). PBSA is an aliphatic polyester resin that has the versatility of common plastic and is stable under ordinary conditions. However, PBSA becomes degradable in compost, wet soil, fresh water, seawater and activated sludge where microorganisms are present. It will decompose completely into water and carbon dioxide, and is therefore called Eco–friendly material (SHOWA DENKO K.K., 2010). Its melting point is 95°C and melt flow rate (MFR) is from one to three (Ikada, 1999a). The reason the PBSA was used is that this biodegradable resin has low modulus and fast biodegradability (Okino, 2005). PBSA that we used is shown in **Photo 4.1**.

TERRAMAC TE-7300, which was developed by Unitika Limited, Japan. TERRAMAC is a biomass material created from plant-derived polymers; the polylactic acid (PLA) that makes up the basis of TERRAMAC is generated from corn, a raw ingredient harvested in large quantities every year. PLA is an aliphatic polyester resin. Its melting point is 170°C and MFR is 17 (Unitika Limited, 2014). PLA used in this study is shown in **Photo 4.2**.

The biodegradable resin, the fine sand, the coarse sand and the calcium carbonate were used to make specimens. The composition of the biodegradable resin concrete is shown in **Table 4.1**. Calcium carbonate is used for compensating for fine-grain fraction. It does not react chemically with biodegradable resin.

Two types of specimens, which have different molecular weights, were made in order to clarify the rate of deterioration by the difference of molecular weight. The weight-average molecular weight (Mw) of these specimens is about 25,000 (low-molecular weight) and 30,000 to 40,000 (high-molecular weight). The molecular weight of former PBSA is very high, it is from 200,000 to 250,000. However, a previous study said that Mw becomes approximately 55,000 after PBSA is heated up for four



A Photo 4.2 PLA

	ratio of mass (%)			
biodegradable resin	12	10		
calcium carbonate	20	20		
fine sand (fineness modulus: 1.09)	20	20		
coarse sand (fineness modulus: 4.91)	48	50		

 Table 4.1 Mixture proportion of biodegradable resin concrete

hours at around 250°C (Ikada, 1999b). In this study, in consideration of workability of resin and aggregates, the biodegradable resin concretes, which have two types of different molecular weights, were made as the viscosity was confirmed with a stirrer. The target viscosity of high-molecular weight specimens is 4dPa·s at 150°C.

4.2.2 Soil to Expose Specimens

Five materials were used to make soil to expose specimens. They were the washed sand, the ligneous compost, the bamboo charcoal, the chicken droppings and the silica sand.

a) Washed sand

The washed sand was used in this study is shown in **Photo 4.3**. It was obtained from Harihara town, Seto city in Aichi prefecture. The properties of the washed sand







Photo 4.3 Washed sand

Photo 4.4 Ligneous compost

Photo 4.5 Bamboo charcoal



and the grain size accumulation curve are shown in **Table 4.2** and **Figure 4.1**, respectively.

b) Ligneous compost

The ligneous compost shown in **Photo 4.4** is manufactured by Gifu ouyou sizai co. ltd. **Table 4.3** summarizes the composition of the ligneous compost. This ligneous compost is composed of bark compost and peat moss and naturally full of bacteria and microbes.

c) Bamboo charcoal

The bamboo charcoal shown in **Photo 4.5** is made of bamboo obtained from Mishima city in Shizuoka prefecture. The properties of the bamboo charcoal are shown in **Table 4.4**. The reason bamboo charcoal was used was to find an efficient use of bamboo, which reflects the increased number of unkempt bamboo forests in Japan and relevant threats involving landslide and groundwater occurrence. Furthermore, it is a

	bark compost	peat moss
mixture proportion (%)	85	15
amount of moisture (%)	67	54.6
pH	6.1	3.8
electric conductivity (ds/m)	-	0.16
organic matter content (%)	91.7	98.65
total nitrogen [N] (%)	2.3	0.67
total phosphoric acid [P2O5] (%)	0.45	0.03
total potassium oxide [K2O] (%)	0.3	0.01>
humus acid (%)	-	9.11
cation exchange capacity [CEC]	85 meq/100g	138.2 cmolc/kg
carbon to nitrogen ratio (%)	20	-

Table 4.3Properties of ligneous compost

Table 4.4 Properties of bamboo charcoal								
production temperature (°C)	850							
specific gravity (g/cm ³)	0.26							
grain size (mm)	1.0~3.0							
carbon (%)	86							
potassium(%)	0.6							
sodium(%)	0.01							
calcium(%)	0.05							
magnesium(%)	0.15							
ferrum(%)	0.01							
manganese (%)	0.5							
silicon (%)	0.6							
germanium (%)	0.05							
ash percentage (%)	2.5							

well-known fact that bamboo charcoal is porous (Hattori and Hosaka, 2007), so we used the bamboo charcoal in this study so that bacteria would be able to live in its pores.

d) Chicken droppings

The chicken droppings used in this study are shown in **Photo 4.6**. Chicken droppings are also full of bacteria and microbes, similar to the ligneous compost. It is used to clarify the difference from ligneous compost. **Table 4.5** shows typical examples of properties of the chicken droppings (Ministry of Agriculture, Forestry and Fishes, 2014).

e) Silica sand

The silica sand used in this study is shown in **Photo 4.7**. The properties and the accumulation curve are shown in **Table 4.6** and **Figure 4.2**, respectively. The silica sand was used to understand the degree of impact of water amount on the biodegradable resin concrete.



Photo 4.6 Chicken droppings



Photo 4.7 Silica sand

Table 4.5 Typical examples of properties	es of chicken droppings
amount of moisture (%)	26
total nitrogen (%)	3.6
phosphoric acid (%)	2.9
potassium oxide (%)	2.0
carbon (%)	31
carbon to nitrogen ratio (%)	9.0
calcium (%)	3.4
magnesium (%)	0.8



4.3 Procedure of Making Specimens

The specimens were fabricated by plate type steel mold and the forming was performed at 200°C. The plate sides are $250 \times 300 \times 30$ mm and $250 \times 300 \times 40$ mm. They were demolded after cooling to 20°C. The procedure of making specimens is shown in **Figure 4.3**. The biodegradable resin concrete plates for the amount of two years were buried. Every couple of months, the plates were dug out and cut.

4.4 Exposure Conditions

Nine patterns of the exposure conditions were treated to clarify the effects of exposure conditions. Adopted exposure conditions are shown in **Table 4.7**. These exposure conditions were put into the plastic boxes, which have 450 mm width, 600 mm length and 350 mm height. In the laboratory, the boxes were kept at approximately

20°C.

This experiment was started from Case A, B and C. Case A and Case B included the ligneous compost. These were made with consideration for a high microorganism environment. Additionally, Case A included the bamboo charcoal, which was mixed to breed microorganisms, while Case C was only sand. This was made with consideration for a low microorganism environment. Case D included the chicken droppings in order to compare the effect of the difference of compost on Case B.

The other exposure conditions (from Case E to Case I) were conducted to clarify the rate of degradation by a difference in water content. Case E; Aridity condition was that specimens were put into the box filled up with silica gel for drying purposes.

The biodegradable resin concretes made from PBSA were exposed in Case A, B, C, and E to I. PBSA specimens were not buried in Case D. On the other hand, the biodegradable resin concretes made from PLA were buried in Case B, C and D. Exposure period is set to two years for a maximum.



30 (40) mm square

Figure 4.3 Procedure of making specimens

Table	Exposure conditions								
	ratio of mass (%)								
	Α	В	С	D	Е	F	G	Η	Ι
washed sand	80	85	90	85	-	-	-	-	-
bamboo charcoal	5	-	-	-	-	-	-	-	-
ligneous compost	5	5	-	-	-	-	-	-	-
chicken droppings	-	-	-	5	-	-	-	-	-
silica sand	-	-	-	-	0	95	90	80	0
water	10	10	10	10	0	5	10	20	100

4.5 Outline of Experiments

4.5.1 Water Absorbency Test

The water absorbency tests were conducted to clear the absorbability of biodegradable resin concrete. The specimens were cubes of 20, 30, 40 mm on each side (**Photo 4.8**). The number of specimens was three for each specimen size. The initial mass of specimens was measured before putting it into water, and was kept under the condition of water temperature at approximately 20°C. The specimens were removed from water and left to stand for approximately two hours for the specimens to dry. After that, the mass of specimens, water temperature and pH were measured.

The soaking periods were from one week through eight weeks. The plastic boxes were refilled with soaking water when we measured specimens.

The water absorbency test of only the resin was conducted. The results are attached as the appendix. (**Photo A-1 and Figure A-1**)

4.5.2 Comparison of Surface Deterioration (Binarization)

The photos of specimens exposed to Case A, B, C and D were taken before mechanical tests. Furthermore, the surface of specimens exposed under Case B, E and Case I were observed with the digital microscope (AnMo Electronics Corp. AM413ZT Dino-Lite Pro Polarizer, **Photo 4.9**). The photos were taken every two weeks, and the shooting points were marked on the specimens to take photos at the same position. Pixels of 1024×1280 were captured and the central portions were cropped in the size of 512×640 . The pixels of 512×640 were binarized at a threshold of 128 after 256 shades of gray images were made (Okuno et al., 2011). The threshold of 128 was determined through trial and error, due to no discrimination of tinted aggregates from resin at threshold of 100 and darker-shaded resin with aggregates at threshold of 150 as seen in **Photo 4.10**. This binarization was conducted for both PBSA specimens and PLA specimens at the same conditions.



Photo 4.8 Specimens of water absorbency test (20, 30, 40mm cube)



Photo 4.9Digital microscope



unprocessed picture







threshold of 150

4.5.3 Microbial Biomass Carbon measurement

Microbial biomass carbon was measured in order to clear the relationship between degradation of specimens and types of soil. Soil samples were collected from plastic boxes every two months. The method was carried out using a similar method to that of Jenkinson and Powlson's fumigation technique (1976). Microbial biomass carbon is calculated from the results of total organic carbon (TOC) of both fumigated soil and non-fumigated soil. In short, the way of getting TOC from fumigated soil is as follows. Moist soil (20 g) was fumigated with purified CHCl₃ for 24h, the CHCl₃ was removed, extracted with 0.5 mol of K₂SO₄ (80 ml), and placed on a shaker for 30 minutes at 200 rpm. The extraction liquid gathered through filter paper No.617_{1/4} was measured by TOC-VCPH (Shimadzu Corporation). The method of infering TOC from non-fumigated soil is the same process as the process of fumigated soil after fumigated. TOC is given by the equation

$$C = \frac{(S-B) \times (E+W)}{SD} \tag{4.1}$$

where *C*: K_2SO_4 -extractable TOC in soil (mg/kg), *S*: analysis value of K_2SO_4 -extractable NPOC (non-volatility organic carbon) in soil (mg/L), *B*: analysis value of K_2SO_4 -extractable NPOC in blank (mg/L), *E*: volume of 0.5 mol K_2SO_4 -extract (ml), *W*: water in soil (ml) and *SD*: dry weight of extracted soil (g)



Figure 4.4 Method of microbial biomass C measurement

Micro biomass carbon is calculated using each result of TOC and the following:

$$C_{mic} = \frac{(C_{fum} - C_{non-fum})}{kC}$$
(4.2)

where C_{fum} is carbon, which is extracted by K₂SO₄ from fumigated soil (mg/kg), $C_{non-fum}$ is carbon, which is extracted by K₂SO₄ from non-fumigated soil (mg/kg), C_{mic} is microbial biomass carbon (mg/kg) and kC is 0.45 (empirical conversion factor, Wu et al., 1990).

A series of the methodology is shown in **Figure 4.4**.

4.5.4 Bending test

The specimens were of two types 30 or 40 mm wide, 30 or 40 mm high and 160 mm long (**Photo 4.11**). The number of specimens was five for each soil type and specimen type. The bending test specified by Japanese Industrial Standards Committee (2006a) was conducted and the bending strength was given by the equation

$$f_b = \frac{3pl}{2bh^2} \tag{4.3}$$

where f_b is the bending strength (N/mm²), p is the maximum load (N), l is the support span (mm), b is the width of specimen (mm) and h is the height of specimen (mm).



Photo 4.11 Bending test

4.5.5 Compressive test

The shape of the specimens tested were cubes of 30 or 40 mm length (**Photo 4.12**). However, generally, it is said that the height of the specimen should be about twice the diameter when a compressive strength testing of concrete is conducted (Nakagawa et al., 2010). In this study, the specimen used was of a cubic shape. This was used to assess the influence of the height/thickness ratio of the specimen on compressive strength to the biodegradable resin concrete. Thus, the pre-compressive test was conducted by changing this ratio. As a result, the influence of the height/thickness ratio on compressive strength was found to be insignificant when compared with commonly-used cement concrete. Therefore, we used cubical specimens for the compressive test. The compressive test specified by Japanese Industrial Standards Committee (2006b) was conducted and the compressive strength was given by the equation

$$f_c = \frac{P}{A} \tag{4.4}$$

where f_c is the compressive strength (N/mm²), p is the maximum load (N) and A is the loading area (mm²).



Photo 4.12 Compressive test

4.6 Results and Discussion

4.6.1 Surface Degradation of Specimens

The degree of surface degradation of the PBSA specimens is shown in **Photo 4.13**. They are high-molecular weight specimens. These specimens degraded with age gradually. Especially, the specimens exposed in the Case B were degraded more remarkable than others. The surface deterioration became pronounced about four months later and the aggregates were exposed. The specimens exposed in Case C degraded slowly, presumably due to contain no ligneous compost. In comparison with the degradation of the specimens exposed in the Case A and B, the specimens exposed in Case B were more remarkable than Case A. In other words, it is suggested that deterioration of biodegradable resin concrete has a pronounced tendency without bamboo charcoal. The reason will be discussed later along with other test results. The surface deterioration was not remarkable for specimens with two months of exposure, whereas, microbial colonized its surface. Therefore, deterioration can be expected to start from at least two months-later in this biodegradable resin concrete.



Photo 4.13 Degradation of PBSA specimens

Furthermore, it has been shown that the surface deterioration of low-molecular weight specimens was more noticeable than these specimens shown in **Photo 4.13**. **Figure 4.5** shows mass change ratio over time. This changes with the passage of time have a strong correlation with the surface deterioration. The mass changes are referred from microbe decomposition. In Case C, the mass change ratio has not changed presumably due to a lack of ligneous compost.

On the other hand, PLA specimens (**Photo 4.14**) provided a slight difference of surface deterioration. From this result, the PLA specimens are less influenced by exposure conditions.





Photo 4.14 Degradation of PLA specimens

4.6.2 Binarization

The micrographs of specimen surface are shown in **Photo 4.15** and the proportions of pixel count of white color to total pixels after binarization are shown in **Figure 4.6**. In Case E; the aridity condition, both PBSA and PLA specimens remained in about the same initial state. In other words, the deterioration may not progress in air-dried state. The specimen showing the most change is the PBSA specimen exposed in Case B. This specimen turned white after two weeks. After that, its surface slowly became black. This means aggregates outcrop due to degradation of resin. However, no PLA specimens in Case B became black, indicating that no surface deterioration occurred. The reason why PLA specimen surface is not deteriorated is said to be that PLA is hydrolyzed without direct involvement of microorganisms (Kimura et al., 2002). The aqueous exposure PBSA and PLA specimens (Case H) did not become black as well. It is inferred from the observation of specimen surface that microorganism in soil decompose PBSA specimens.



Photo 4.15 Micrograph of specimens surface (PBSA and PLA)



4.6.3 Water Absorbency Test

Table 4.8 shows the condition of soaking water. According to **Table 4.8**, the pH value rose a little after soaking specimens. This elevated pH has a negligible effect on surrounding environment (Ministry of Health, Labour and Welfare, 2014). The absorbability of biodegradable resin concrete is shown in **Figure 4.7**. The absorbability of biodegradable resin concrete was stopped in about four weeks. The saturation velocity was similar regardless of the size of specimen. Absorbability of PLA in comparison with PBSA was high. This results prove that PLA has high reactive with water as is the case with surface deterioration (Kimura et al., 2002).

Table 4.6 Conditions of soaking water										
	initial	1 day	1 week	2 weeks	3 weeks	4 week	s 5 weeks	6 weeks	7 weeks	8 weeks
water temperature (°C)	21	19.8	20	19.8	19.8	20.1	20	20	20.2	20.2
water pH soaked PBSA	7.2	8.2	8.2	8	8.2	8.2	8.1	8.1	8.5	8
water pH soaked PLA	7.2	7.8	7.9	7.9	8.1	8	8	7.9	7.9	7.9
	1.0 0.9 0.8 0.7 0.5 0.5 0.5 0.4 0.3 0.2 0.1		40mm 30mm 20mm				PBSA			
	0.0	•	· .		. 1.		🛓 .	<u> </u>		
	0.0	1	2	34 aking r	5 eriod (w	67 veek)	8	9		
ē	1.0 0.9 0.8 0.8		40mm 30mm 20mm				PLA	-		
	0.4 0.3 0.2 0.1 0.0		• • 2			6 7				
	soaking period (week)									

 Table 4.8
 Conditions of soaking water

Figure 4.7 Absorbability of specimens (PBSA and PLA)

4.6.4 Bending Test

The bending test results of PBSA are shown in Figure 4.8. A plot in the figure is the mean of five specimens. The difference in color of the plot expresses the difference of the soil to exposed specimens. The bending strength was reduced in all specimens with different resin ratio, thickness and soil pattern. The bending strength decreases remarkably for a period of six months after exposure. This strength reduction will not be caused microorganism, because microbial degradation speed is slow. In fact, the rate of mass change has not decreased (Figure 4.6). Therefore, this remarkable strength reduction may be caused from water. The bond strength between biodegradable resin and aggregates may be reduced because that the exfoliation of aggregates from the resin was confirmed at the broken-out section after bending test. Furthermore, two-phased strength reduction is shown in this figure. The rate of strength reduction changed in two months exposure. This two-phased decomposition process is normally found in biodegradable plastics. For a month or two after burying the soil, it is said that a hydrolysis reaction occurred and molecular weight is reduced to lower than 20,000 (Kitamoto, 2009). Therefore, it is suggested that the biodegradable resin concrete used in this study had the same reaction as in the resin plastics.

From the above discussion, water affected the strength reduction shown during the early exposure period, and led to gradual strength reduction shown after several months of exposure due to microbial degradation.

The comparison of bending strength due to the difference of molecular weights is shown in **Figure 4.9**. It is obvious that the difference of the molecular weight depends exclusively on early-age strength. It is an acceptable result that the rate of decrease in the bending strength of the lower molecular weight specimens was small, if a hydrolysis reaction occurred and the molecular weight was reduced to lower than 20,000 for the age of a month or two as previously mentioned.



Figure 4.8 Bending test results of PBSA (30mm thick and 40mm thick)



Figure 4.9 Comparison of bending strength due to differences in molecular weight

Figure 4.10 shows the strength retention by a variation in water content. The absorbability of this biodegradable resin concrete was stopped in about four weeks as described above, and the remarkable reduction was stopped in about six months. Therefore, the effect of water on the biodegradable resin concrete will occur a few months after exposure. This is the reason this experiment was terminated in order to clarify the bending strength reduction by a variation in water content up to four months. The specimens of atmospheric curing (Case E) have little deterioration. The tendency of strength reduction from Case E to Case I was similar. From this result, this biodegradable resin concrete can be degraded if it is exposed in soil contained over 5% water.



Figure 4.10 Strength retention by a variation in water content

Figure 4.11 shows the bending test results of PLA. A plot in the figure is the mean of five specimens. The difference in color of the plot is the difference between the soil and the exposed specimens. The bending strength was reduced in all specimens with different resin ratios, thicknesses and soil patterns. Two-phased strength reduction as PBSA specimens were not shown. The bending strength of PLA specimens declined in a linear manner. The difference of downward trend in bending strength is obvious from **Figure 4.12**. The difference of strength reduction tendency will be caused by the difference of chemical bonding. PBSA consists of condensation polymerization of glycol and dicarboxylic acid. On the other hand, PLA consists of ester binding of acidum lacticum.



Figure 4.11 Bending test results of PLA (30mm thick and 40mm thick)



Figure 4.12 Comparison of bending strength due to differences in resin (PBSA and PLA)

4.6.5 Compressive Test

The compressive test results are shown in **Figure 4.13**. The compressive strength reduction was observed in all specimens with different resin ratios, thicknesses and soil patterns. Compressive strength stems largely from the strength of the aggregates; on the other hand, bending strength is the ascribable tensile strength of resin. This is because the residual strength was kept higher than bending residual strength. In fact, the deterioration has not progressed into the middle of specimens as shown **Photo 4.16**. In other words, the inner part of the specimen has retained strength. There was little difference between soil types and both deterioration of the bending strength and the



compressive strength. As well as bending strength reduction, the specimens buried in Case C had gradual degradation similar to others.

Figure 4.13 Compressive test results of PBSA (30mm thick and 40mm thick)



Photo 4.16 Cross-section surface of the specimen

The comparison of compressive strength due to differences in molecular weight is shown in **Figure 4.14**. It is found that the difference of the molecular weight depends exclusively on the early-age strength. However, the tendency is less apparent than in the bending test. As mentioned before, the results mean that compressive strength stems largely from the strength of the aggregates.



Figure 4.14 Comparison of compressive strength due to differences in molecular weight

Figure 4.15 shows the compressive test results of PLA. A plot in the figure is the mean of five specimens. The difference in color of the plot is the difference between the soil and the buried specimens. Compressive strength was reduced in all specimens with different resin ratios, thicknesses and soil patterns. The early-age compressive strength of PLA was much higher than in PBSA. PLA specimens showed compressive strength three to four times higher than in PBSA specimens. These results made it clear that PLA has a high tolerance for compression.



Figure 4.15 Compressive test results of PLA (30mm thick and 40mm thick)

Figure 4.16 shows compressive strength due to differences in resin. As mentioned before, the early-age compressive strength of PLA was very different from PBSA. The compressive strength of PBSA was approximately 25 N/mm²; on the other hand the compressive strength of PLA was approximately 70 N/mm². However, the tendency of strength reduction was similar.



Figure 4.16 Comparison of compressive strength due to differences in resin (PBSA and PLA)

4.6.6 Results of Microbial Biomass Carbon Measurements and Microbial Identification

The measurements of microbial biomass carbon are shown in **Figure 4.17**. Microbial biomass carbon increased a little bit after two months of curing. It is suggested that strength reduction was also due to microbial decomposition. Case C did not contain ligneous compost, so microbial biomass carbon was lower than Case A and Case B. The rate of strength degradation of specimens exposed in Case C was gradual. We used bamboo charcoal in expectation that bacteria were able to live in the pore. Nevertheless, the microbial biomass carbon was lower than Case B. The result leads to our presumption that the multiplication of the microorganism was false.



Figure 4.17 Microbial biomass C (PBSA and PLA)

The results on the amount of microbial biomass carbon in this study are not only polybutylene succinate adipate (PBSA) degrading bacteria but also the amount of total organic carbon. Therefore, it is necessary to identify the degrading bacteria of this biodegradable resin concrete. From the results of genomic analysis, PBSA degrading bacteria are Fusarium solani, Streptomyces albogriseolus, Ochroconis constricta and Gliomastic Murolum. It was clarified that these bacteria are aerobic bacteria. The certificates of microbial analysis are attached as the appendix.

From the tendency of increasing and decreasing of microbial biomass carbon, it is inferred that the effect of population density had occurred. The exposure environment of this study is not of a natural condition, but of a closed condition. Therefore, there is some possibility that microbial will be increased and the degradation of specimens will be faster if the biodegradable resin concrete is exposed in natural conditions.



Photo 4.17 PBSA degrading bacteria

4.7 Practical Application

4.7.1 Introduction

The final goal of this study is to achieve the practical use of biodegradable resin concrete. Conceivable applications are primarily piles, sheet piles, temporary roads, slope face stabilizers and pipelines. As mentioned above, the mechanical properties and tendency to deteriorate of biodegradable resin concrete specimens was clarified.

In this section, some mechanical test results of biodegradable resin concrete pipe were described as a step toward the practical use. Biodegradable resin concrete pipes are expected to contribute to trenchless technology as shown in **Figure 4.18**. A jacking method using a biodegradable resin concrete pipe is expected to largely contribute to removing an existing pipe.



(Reference: Kizar fecto Corp. 2014)

Figure 4.18 Application example of biodegradable resin concrete pipe

4.7.2 Procedure of Making Biodegradable Resin Concrete Pipe

The procedure of making biodegradable resin concrete pipe is shown in Photo

- **4.18**. The process flow is given below.
- 1. Melt PBSA resin by heating.
- 2. Heat aggregates and a blender to 180 degree.
- 3. Put melted PBSA resin into the blender.
- 4. Mix the resin and the aggregates well.
- 5. Put the mixture into a turning metallic mold; centrifugal machine.
- 6. Keep 30 minutes the centrifugal machine with the same temperature and rate of rotation.
- 7. Cooling with turning the machine.
- 8. Demolded.



Photo 4.18 Procedure of making biodegradable resin concrete pipe

4.7.3 Out Line of Experiments

The dimensions of the biodegradable resin concrete pipes used in circumferential compression test are $\phi 360 \times 200$ mm and 30 mm thickness. The appearance of the pipe is shown in **Photo 4.19**. The dimensions of the pipes used in axial compression test are $\phi 360 \times 600$ mm and 30 mm thickness.

In the case of the circumferential compression test, four strain gauges were attached on the interior surface and two strain gauges were attached on the exterior surface. In the case of the axial compression test, for strain gauges were attached on exterior surface (**Figure 4.19**). The number of specimen pipes was three for each soil.



Photo 4.19 Appearance of biodegradable resin concrete pipe



Figure 4.19 Attached position of strain gauges

4.7.4 Test Results and Discussion

a. Initial Strength

Figure 4.20 shows the axial compression test results, and **Figure 4.21** shows the circumference compression test results. These strength results are the initial pipe strength. The first pipe was of lesser strength than others because inexperience in making the sample. However, axial compression strength showed approximately 700 kN (21 MPa). This axial compression strength is enough to shove and remove an existing pipe. At the end of this thesis, the photos of the situation of experiment and the destroyed pipes are attached.



Figure 4.21 Circumferential compression test result (initial strength)

b. Strength Reduction with Exposure Period

Figure 4.22 and Figure 4.23 show time degradation of biodegradable resin concrete pipes. The axial strength of this pipe had high strength, which is over 12 kN/mm^2 for one year after exposure. On the other hand, the circumferential strength decreased to around 1 kN. From this result, a land where the biodegradable resin concrete pipe is buried can be redeveloped easily because the bearing force from above is low.

Two kinds of soil were used as filled material into the pipe (Case C and Case D). However, it is clarified that the difference of filled materials had no appreciable effect on strength.



Figure 4.23 Circumferential compression test result

4.8 Conclusions

The degradation level was clarified by a mechanical approach to biodegradable resin concrete. The biodegradable resin concrete is a new product, and it is different from usual cement concrete. It is considered that the most important thing about the commonly-used cement concrete is to enhance strength. On the other hand, biodegradable resin concrete has strength or stiffness deterioration properties. Therefore, we conducted bending test and compressive test mainly to clarify the degradation level of the biodegradable resin concrete. Furthermore, the biodegradable resin concrete pipe was created and mechanical tests were conducted to work towards practical use. From these results, the following conclusions were obtained.

- 1. The absorbability of biodegradable resin concrete was stopped in approximately four weeks.
- 2. The surface degradation became pronounced about four months later and the aggregates were exposed. At two months exposure, microbial colonized its surface.
- 3. The rate of mass changes exhibits a correlation with the degree of degradation of specimens.
- 4. The difference of molecular weight of the biodegradable resin concrete depends exclusively on early-age strength. For a month or two after burying in the soil, a hydrolysis reaction occurred and molecular weight was reduced to lower than 20,000 as the same as resin plastics. Therefore, the two-phased decomposition processes were shown in the results of bending test and the compressive test.
- 5. The bamboo charcoal may not be suitable for degrading this biodegradable resin concrete and the ligneous compost need to use in order to accelerate degradation of this biodegradable resin concrete.
- 6. The effect of water on the biodegradable resin concrete will occur a few months after exposure
- 7. The bending strength decreases remarkably for a period of six months after exposure. The remarkable strength reduction may be caused not by microorganism but by
water. The bond strength reduction between resin and aggregates and a hydrolysis reaction occurred by water.

- This biodegradable resin concrete can be degraded if it is exposed to soil contained over 5% water.
- 9. The residual compressive strength on initial strength was kept higher than residual bending strength.
- 10. The microbial biomass carbon repeatedly fluctuated because of the effect of population density. However, the soil containing bamboo charcoal had a smaller amount of microbial biomass carbon in virtually exposure period. Bamboo charcoal cannot be used as the breeding material for microorganisms.
- 11. PBSA degrading bacteria are Fusarium solani, Streptomyces albogriseolus, Ochroconis constricta and Gliomastic Murolum. These three types of bacteria are aerobic bacteria.
- 12. The biodegradable resin concrete pipe was of adequate initial strength. These test results provide the possibility of being helpful in the jacking method without depending on an open-cut method.
- 13. After exposure of biodegradable resin pipes, both axial strength and circumferential strength decreased gradually. At the present time, a suitable filled material has yet to be found. However, the adequate strength reduction was shown even when only sand, which was made with consideration for low microorganism environment, was used as the filled material.

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CHAPTER 5

The contents of this chapter are based on:

Suzuki, M., Kubo, K. and Kawabata, T. (2014): Statistical Estimation Approach of Degradation of Biodegradable Resin Concrete, *Concrete Research and Technology*, (in Japanese) (submitted)

Chapter 5

Estimation Approach of Degradation of Biodegradable Resin Concrete

5.1 Introduction

The final goal of this study is aimed towards applying biodegradable resin concrete to temporary materials in the future. Temporary materials have to maintain adequate strength during a certain construction period. It is very important to estimate fatigue life and to reveal how long the temporary materials application of biodegradable resin concrete retain the expected strength.

In the previous chapter, some mechanical test results were described, and the degradation characteristics and the degrading factors of biodegradable resin concretes were revealed. In this chapter, model equations of deterioration prediction of biodegradable resin concrete by means of the results are described.

5.2 Degradation Factor of Biodegradable Resin Concrete

According to JIS K 6900, 'degradation' means 'the changes in the chemical structure of a harmful alteration to the characteristics'. In this study, 'degradation' can be defined as all changes including changeless of chemical structure such as resin expansion. The degradation factor of biodegradable resin concrete is assumed to have physical, chemical and biological factors because biodegradable resin concrete is made from high-polymer material (Ishikawa and Tomioka, 2005). Specifically, the physical factor is the expansion of biodegradable resin, while the chemical factor is hydrolysis and the biological factor is microbial degradation. There are more degradation factors such as temperature, light and ultraviolet (Honma, 2005). However, the effect of these degradation factors on biodegradable resin concrete has been unidentified.

5.3 Estimation Models of Degradation

There are some prediction methods of degradation. For example, there are physical and chemical formulas, statistical formulas, and the way of turning numbers into an easily existing formula (Nishimura et al, 2010). Physical and chemical formula can be deduced based on the degradation mechanism (Takahashi, 2004). Using physical and chemical formulas is ideal for the prediction method of degradation. However, it is hard work to pin down the physical and chemical factors. Typically, the cause of degradation is complexly intertwined with related factors. The cause of degradation of the biodegradable resin concrete is complex as mentioned in previous chapter. Therefore, statistical formulas are created. Statistical formulas have a number of advantages as estimation models of degradation. First, statistical formulas can include the complex deteriorative process in only one model. Second, statistical formulas can involve variability of the quality of specimens.

There are many kinds of statistical formulas. Above all things, 'Weibull distribution' were chosen. Weibull distribution will be explained in the next section.

5.4 Weibull Distribution

The Weibull distribution was suggested by W. Weibull in 1939 during his experiments of metallic fatigue life (Weibull W. 1951). At that time, he concluded that the breakdown of brittle materials occurred due to independent flaw in materials. The Weibull distribution is one of the most widely used distributions in dependability analysis because it is flexibly applicable to fault curves (Fukui, 2006 and Inazumi, 2013).

5.4.1 Weibull Probability Distribution Function (The 2-Parameter Weibull)

The 2-parameter Weibull probability distribution function is given by

$$F(t)=1-R(t)=1-\exp\left\{-\left(\frac{t}{\beta}\right)^{\alpha}\right\}$$
(5.1)

where R(t): degree of confidence, t: time, α : shape parameter, and β : scale parameter.

The probability distribution function F(t) of the order statistics is given by mean rank. Mean rank is determined by the number of sample and the order. Mean rank has no relationship to the distributed data. The probability distribution function F(t) by mean rank is given by

$$F(t) = \frac{n}{N+1} \tag{5.2}$$

where N: the number of total sample and *n*: the order of data

Eq. (5.1) is expanded to obtain MTTF (mean time to failure) as follows. 1. Take the log of both sides of

$$\ln\left\{\ln\frac{1}{1-F(t)}\right\} = \alpha \ln t - \alpha \ln\beta$$
(5.3)

2. Replace $\ln t$ with X and $\ln \left[-\ln\{1-F(t)\}\right]$ with Y

$$\ln t = X$$
, $\ln [-\ln \{1 - F(t)\}] = Y$ (5.4)

obtained

$$Y = \alpha X - \alpha \ln \beta \tag{5.5}$$

From Eq. (5.5), the regression line is obtained. The average and the standard deviation are calculated using linear regression analysis and mean rank, Eq. (5.2).

$$MTTF = \beta \Gamma \left(1 + \frac{1}{\alpha}\right)$$
(5.6)
$$\sigma^{2} = \beta^{2} \left\{ \Gamma \left(1 + \frac{2}{\alpha}\right) - \Gamma^{2} \left(1 + \frac{1}{\alpha}\right) \right\}$$
(5.7)

where *MTTF* is mean time to failure, σ is standard deviation and Γ is the Gamma function.

5.4.2 Example of the Application of Weibull Distribution

Figure 5.1 shows the relationships between exposure period and bending strength in the biodegradable resin concrete. As an example, the procedure that the probability distribution of the biodegradable resin concrete specimens become under 3 kN/mm² is given.

First, the specimens showing bending strength below 3 kN/mm² are counted. Second, the Weibull probability distribution is derived from Eq. (5.2) and Eq. (5.4). The Weibull probability distribution graph is shown in **Figure 5.2**. Third, α and β are calculated using the slope and intercept getting from Weibull probability distribution plot. Finally, MTTF and σ^2 are calculated from Eq. (5.6) and Eq. (5.7). In this time, MTTF was 19.1 months and σ^2 was 9.3 months.



Figure 5.1 The relationships between exposure period and bending strength



Figure 5.2 Weibull probability distribution (bending strength show below 3kN/mm²)

5.5 Results and Discussion

The MTTF of PBSA is listed in **Table 5.1**. It shows how much time will be required to drop to below each strength. **Figure 5.3** shows the estimation model degradation of bending strength and **Figure 5.4** shows the estimation model degradation of compressive strength. These figures are on the PBSA specimens. It will be quite a long time before the strength of biodegradable resin concrete becomes zero from the present estimation model. This is not a surprising result because biodegradable manufacture in previous study by other researchers retained the shapes and strength even though the ropes were exposed for three years (Nishimura, 2007). However, biodegradable resin concrete will not be harmful to land development because it is expected to reduce strength adequately. From **Figure 5.3**, it was clarified that the difference of molecular weight has relations with residual bending strength. The mean time before the strength reduce below 3kN/mm² is the same of degradation regardless of molecular weight.

PBSA bending strength prediction compressive strength prediction low-molecular weight high-molecular weight low-molecular weight high-molecular weight 16.1 months 13.9 months 15.0 months 14.3 months 4 N/mm^2 5 N/mm^2 12 N/mm^2 17 N/mm^2 14.5 months 26.0 months 19.1 months 4 N/mm^2 6 N/mm^2 15 N/mm^2 15.7 months 3 N/mm^2 2 N/mm^2 25.1 months 3 N/mm^2 20.4 months 4 N/mm^2 30.5 months 12 N/mm^2 19.4 months 48.4 months 23 years 40.9 months $8N/mm^2$ 31.4 months 1 N/mm^2 2 N/mm^2 2 N/mm^2

Table 5.1 Mean time to failure (MTTF) of PBSA



Figure 5.3 Estimation model of degradation (bending strength of PBSA)



Figure 5.4 Estimation model of degradation (compressive strength of PBSA)

Table 5.2, **Figure 5.5** and **Figure 5.6** show the estimation model degradation of PLA. The bending strength prediction curve of PLA is similar to PBSA. For the last time, the bending strength converge 2 kN/mm². On the other hand, the compressive strength prediction curve of PLA is different from PBSA.

Table 5.2 Mean time to failure (MTTF) of PLA						
PLA						
bending stren	gth prediction	compressive strength prediction				
8 N/mm ²	11.9 months	40 N/mm ²	10.5 months			
5 N/mm ²	13.7 months	30 N/mm ²	15 months			
3 N/mm ²	16.6 months	20 N/mm ²	19.4 months			
2 N/mm^2	26.8 months					



Figure 5.5 Estimation model of degradation (bending strength of PLA)



Figure 5.6 Estimation model of degradation (compressive strength of PLA)

5.6 Conclusions

The estimation models of degradation were created using the Weibull distribution. From the estimation model, the following findings were obtained.

- It will be quite a long time before the strength of biodegradable resin concrete become zero from the present estimation model. However, biodegradable resin concrete will not be obstacles to the land development because it is expected to reduce strength adequately.
- The difference of molecular weight has relations with residual bending strength. The mean time before the strength reduces below 3 kN/mm² is the same, regardless of molecular weight.
- The bending strength prediction curve of PLA is similar to PBSA. For the last time, the bending strength converge 2 kN/mm².

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CHAPTER 6

Chapter 6

Conclusions and Perspectives

6.1 Introduction

This study has been conducted as a fundamental study of construction materials for enhancing cycle society. Developing new construction materials is of importance because concern on resource scarcity and environmental pollution has been increasing. In this thesis, the usability of the improved soil using muddy soil and the biodegradable resin concrete have been identified.

The following shows the conclusions of the previous chapters.

6.2 Conclusions and Perspectives

Chapter 2 was focused on the laboratory experiment of improved soil using muddy soil. It was clarified that the improved soil has to be blended after considering the properties water content and grain size distribution. It is necessary that the improved soil be mixed homogeneously with effectual water content and grain size distribution to provide the adequate strength. The long-term strength can be anticipated if the quicklime and the fly ash are added. The quicklime and the fly ash also play a role in reducing coefficient of permeability. It is clarified from the observation with a scanning electron microscope that long-term strength and permeability reduction stem largely from densification by calcium silicate hydrate.

Chapter 3 was focused on the field experiment of improved soil using muddy soil. In this field test, the large soil mixing machine was used. From the field test, it was clarified that the improved soil should be scrambled three or more times to increase strength.

The previously described two chapters are mentioned about improved soil for reconstructing small earth-fill dams. Many old small earth-fill dams are faced with danger from disasters. Therefore, it is necessary to reconstruct them in consideration of the cost and the environment aspect.

The major challenge for this thesis is to create the biodegradable resin concrete. The biodegradable resin concrete is a new construction material, and the biodegradable resin concrete was made through trial and error. The detailed procedure and the properties of the biodegradable resin concrete are mentioned in Chapter 4. The mechanical properties are very different from commonly-used cement concrete. The strength or stiffness of biodegradable resin concrete degrade over time. Especially, the bending strength decreases remarkably for a period of six months after exposure. This strength reduction will not be caused by microorganism. The remarkable strength reduction may be caused by water. The bond strength reduction between resin and aggregates, and a hydrolysis reaction occurred due to water. Furthermore, it was clarified that this biodegradable resin concrete can be degraded if it is exposed in soil contained over 5% water. This means the following; the strength reduction of biodegradable resin concrete must be caused by a small amount water. On the other hand, the surface degradation became pronounced about four months later and the aggregates were exposed. At two months exposure, microbial colonized its surface. Additionally, the rate of mass changes had a correlation with the degree of degradation of specimens. This means the following, the surface degradation without strength reduction must be caused by microorganisms.

The final goal of the study on biodegradable resin concrete is to achieve the practical use. Therefore, the biodegradable resin concrete pipes were made and some mechanical tests were conducted. At the end of chapter 4, the procedure of making the biodegradable resin pipe and the mechanical test results are written. As the result, the biodegradable resin concrete pipe was of adequate initial strength. These test results raise the possibility of being helpful in the jacking method without depending on an open-cut method. After expose, both axial strength and circumferential strength decreased gradually. At the present time, the suitable filled material have yet to be found.

In Chapter 5, the model equations of deterioration prediction of biodegradable resin concrete were created by means of the results of Chapter 4. There are many prediction methods. In this thesis, the estimation models of degradation were created using the Weibull distribution. From the distribution model, it was clarified that it would be quite a long time before the strength of biodegradable resin concrete become zero from the present estimation model. However, biodegradable resin concrete will not be obstacles to the land development because it is expected to reduce strength adequately.

The biodegradable resin concrete need to be studied much further. Biodegradable resin concrete is known to deteriorate with many factors such as microbial action, water, temperature, light and ultraviolet. In this study, the microbial degradation and the action of water have mentioned. Therefore, the effect of other factors on biodegradable resin concrete have to be clarified. If the method to control deterioration is found, the biodegradable resin concrete will be easier to use.

Appendix

Chapter 4 (Experiment of Biodegradable Resin Concrete)

The measuring instrument of only resin expansion is shown in **Photo A-1**. The result is shown in **Figure A-1**.



Photo A-1 Measuring instrument of resin expansion



Figure A-1 Expansion of resin alone (PBSA, Mw=30,000 to 40,000)

The certificates of microbial analysis are shown in From Figure A-2 to Figure A-9.





解析

25 産総食技第1-1003号 別紙

	10	20	30	40	50	60	70
	AAACCAACAG	GGATTGCCCC	AGTAACGGCG	AGTGAAGCGG	CAACAGCTCA	AATTTGAAAT	CTGGCTCTCG
	80	90	100	110	120	130	140
	GGCCCGAGTT	GTAATTTGTA	GAGGATGCTT	TTGGTGAGGT	GCCTTCCGAG	TTCCCTGGAA	CGGGACGCCA
	150	1,60	170	180	190	200	210
	TAGAGGGTGA	GAGCCCCGTC	TGGTTGGACA	CCGATCCTCT	GTAAAGCTCC	TTCGACGAGT	CGAGTAGTTT
	220	230	240	250	260	270	280
	GGGAATGCTG	CTCTAAATGG	GAGGTATATG	TCTTCTAAAG	CTAAATACCG	GCCAGAGACC	GATAGCGCAC
	290	300	310	320	330	340	350
	AAGTAGAGTG	ATCGAAAGAT	GAAAAGAACT	TTGAAAAGAG	AGTTAAACAG	TACGTGAAAT	TGTTGAAAGG
ł	360	370	380	390	400	410	420
	GAAGCGCTTG	TGACCAGACT	TGGGCTTGGT	TGATCATCCG	GGGTTCTCCC	CGGTGCACTC	TTCCGGCTCA
	430	440	450	460	470	480	490
	GGCCAGCATC	AGTTCGCCCT	GGGGGATAAA	GGCTTCGGGA	ATGTGGCTCT	CTCCGGGGAG	TGTTATAGCC
	500	510	520	530	540	550	560
	CGCTGCGTAA	TACCCTGTGG	CGGACTGAGG	TTCGCGCATT			

Figure A-3 Base sequence (Fusarium solani)



Figure A-4 Certificate of microbial analysis (Streptomyces albogriseolus)

戦後に行う

25產総食技第1-1005号 別紙

GGACGAACGC TGGCGGCGTG CTTAACACAT GCAAGTCGAA CGATGAACCA CTTCGGTGGG GATTAGTGGC GAACGGGTGA GTAACACGTG GGCAATCTGC CCTGCACTCT GGGACAAGCC CTGGAAACGG GGTCTAATAC CGGATACTGA CCCGCTTGGG CATCCAAGCG GTTCGAAAGC TCCGGCGGTG CAGGATGAGC CCGCGGCCTA TCAGCTTGTT GGTGAGGTAA TGGCTCACCA AGGCGACGAC GGGTAGCCGG CCTGAGAGGG CGACCGGCCA .340 CACTGGGACT GAGACACGGC CCAGACTCCT ACGGGAGGCA GCAGTGGGGA ATATTGÓACA ATGGGCGAAA GCCTGATGCA GCGACGCCGC GTGAGGGATG ACGGCCTTCG GGTTGTAAAC CTCTTTCAGC AGGGAAGAAG CGAAAGTGAC GGTACCTGCA GAAGAAGCGC CGGCTAACTA CGTGCCAGCA GCCGCGGTAA TACGTAGGGC GCGAGCGTTG TCCGGAATTA TTGGGCGTAA AGAGCTCGTA GGCGGCTTGT CACGTCGGTT GTGAAAGCCC GGGGCTTAAC CCCGGGTCTG CAGTCGATAC GGGCAGGCTA GAGTTCGGTA GGGGAGATCG GAATTCCTGG TGTAGCGGTG AAATGCGCAG ATATCAGGAG GAACACCGGT GGCGAAGGCG GATCTCTGGG CCGATACTGA CGCTGAGGAG CGAAAGCGTG GGGAGCGAAC AGGATTAGAT ACCCTGGTAG TCCACGCCGT AAACGGTGGG CACTAGGTGT GGGCGACATT CCACGTCGTC CGTGCCGCAG CTAACGCATT AAGTGCCCCCG CCTGGGGAGT ACGGCCGCAA GGCTAAAACT CAAAGGAATT GACGGGGGGCC CGCACAAGCG GCGGAGCATG TGGCTTAATT 940 -CGACGCAACG CGAAGAACCT TACCAAGGCT TGACATACAC CGGAAACGTC CAGAG

Figure A-5 Base sequence (Streptomyces albogriseolus)



Figure A-6 Certificate of microbial analysis (Ochroconis constricta)

25產総食技第1-1007号 別紙

	10	20	30	40	50	60	70
	TAACGGCGAG	TGAAGCGGCA	ACAGCTCAAA	TTTGAAATCT	GGAGCCTTTG	TTGGCTTCCG	AGTTGTAATT
	80	90	100	110	120	130	140
	TGCAGAGGGA	GCCTCGGAGG	GCTGGTCGGT	CCAAGTGCCT	TGGAACAGGA	TACCGCAGAG	GGTGAGAGTC
	150	160	170	180	190	200	210
	CCGTATCCGG	CCGACGCAGC	GCTCCGTGTG	AGGCCTCTTC	GACGAGTCGA	GTTGTTTGGG	AATGCAGCTC
	220	230	240	250	260	270	280
	TAAGTGGGTG	GTAAATTCCA	TCTAAAGCTA	AATACTGGCC	AGAGACCGAT	AGCGAACAAG	TAGAGTGATC
	290	300	310	320	330	340	350
	GAAAGATGAA	AAG				1	0.000
a							



Figure A-7 Base sequence (Ochroconis constricta)



Figure A-8 Certificate of microbial analysis (Gliomastic Murolum)

25産総食技第1-1259号別紙

品名:生分解レジンコンクリートより採取した微生物K3 AACCAACAGG GATTGCCTCA GTAACGGCGA GTGAAGCGGC AACAGCTCAA ATTTGAAATC TGGCCTCTGG CCCGAGTTGT AATTTGCAGA GGATGTTTCT GGCGAGGTGC CTTCCGAGTT CCCTGGAACG GGACGCCATA GAGGGTGAGA GCCCCGTACG GTTGGTCGCT AAGCCTCTGT GAAACTCCTT CGACGAGTCG AGTAGTTTGG GAATGCTGCT CTAAATGGGA GGTGTACGCC TTCTAAAGCT AAATATAGGC TAGAGACCGA TAGCGCACAA GTAGAGTGAT CGAAAGATGA AAAGCACTTT GAAAAGAGGG TTAAATAGTA CGTGAAATTG TTGAAAGGGA 楽皿ンピ AGCGCTCTTS ACCAGACTTG CGCCGGTTGA TCATCCACCG TTCTCGGTGG TGCACTCTGC CGGCTCAGGC CAGCATCAGT TCGGCTTGGG GGATAAAGGC TTCGGGAATG TGGCTCTCTC CGGGGAGTGT TATAGCCCGT TECETAATAC CCTEGECCEE ACTEAGETTC GCCCATCTEC ATEGATECTE SCETAATEST CATCAETEAC CCGTCTTGAA ACACG

Figure A-9 Base sequence (Gliomastic Murolum)

Chapter 6 (planed practical application of Biodegradable Resin Concrete)



The photos of biodegradable resin concrete pipe

Photo A-2 Biodegradable resin concrete pipe before experiment

Appendix



Photo A-3 Biodegradable resin concrete pipe after experiment (axial compression test)



Photo A-4 Biodegradable resin concrete pipe after experiment (circumferential compression test)

A Study on Mechanical Properties of New Environmentally-friendly Construction Materials (環境に配慮した新しい建設材料の力学特性に関する研究)

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農業と環境には強い結びつきがあり,生産性を高めるために技術革新が行なわれる一方で,環境 に負荷を与えていた. 1980年代後半から環境問題に対する意識が高まり,近年,農業分野でも'持 続可能な農業'という考えに注目が集まっている.

本研究では、環境に配慮した新しい土木材料の力学特性に関する研究と題し、農業工学分野にお ける環境問題へのアプローチを試みた.博士論文は二部構成(全6章)になっており、第一部(2 章、3章)では、ため池汚泥を再利用した改良土に関する研究、第二部(4章、5章)では、生分解 性樹脂コンクリートに関する研究について述べる.各章ごとの要旨を以下の通りである.

第1章では、近年の環境についての問題を提起し、既往の研究の概観を試みるとともに、本研究 の位置づけや目的について説明した.

第1部 ため池汚泥を用いた改良土に関する研究

ため池は食糧供給において重要な役割を担っており、古くから多く構築され、現在、全国に約21 万個ため池が存在している.特に、兵庫県下では、全国で最も多い、約4万4千個のため池があ る.これら多くのため池が老朽化という問題を抱えており、地震や台風などによって大きな被害を 受けている.そこで早急な改修が必要であるが、ため池改修を実施する上で、適切なコア用土の確 保が課題として存在する.また、築造年代が古く老朽化したため池には、底泥土が堆積しており、 悪臭や貯水量低下の原因となっている.このようなため池を取り巻く負の環境を解決するために、 環境に優しく安価で力学的に安定した改良土の創出を目標に、現場発生土である底泥土と旧堤体盛 土材の再利用を検討した.

第2章では、底泥土と旧堤体盛土材の適切なブレンド割合を明らかにするために、様々な割合で 作製された改良土に対して一軸圧縮試験や透水試験などを実施するとともに、生石灰や産業廃棄物 であるフライアッシュの混和材としての有用性を検討した.また、SEMによって改良土の構造を微 細に観察した.各試験の結果、底泥土と旧堤体盛土材の改良土がため池コア用土として十分な強度 と遮水性を有するためには、適切な細粒分の割合を有し、最適な含水比で均質に混合されることが 必要であることがわかった.また、生石灰やフライアッシュを混合するとケイ酸カルシウム水和物 が生成され緻密構造になり、更に大きな強度が発現されることが SEMにより明らかとなった.

第3章では、実施工を考慮し、均質材料が必要なコア用土作製に、大型混練機を用い現場作製し た混合土のバラツキを評価し現場への適用性を検討した。その結果、混練回数を増加させることに よって、一軸圧縮強度増加が見られた。また、バラツキが小さくなることも明らかとなった。よっ て、均質な試料が必要なため池コア用土作製に、大型混練機を用いる場合、1回混練ではなく3回 以上混練することが不可欠であることがわかった。
第2部 生分解性樹脂コンクリートに関する研究

施工時に使用された矢板などの仮設資材は,通常,施工完了後に引抜き撤去されるが,撤去時に 近接構造物への影響が懸念される場合など,安全性を考慮して残置されるケースが増加している. しかしながら,地中に残存された不要構造物は,再開発時に障害物や廃棄物となり,土地の有効利 用上の阻害要因に繋がる可能性がある.このような社会的背景を受け,骨材と微生物によって分解 可能な樹脂からなる生分解性樹脂コンクリートを矢板や杭材などの仮設資材に適用することを考案 した.

第4章では、生分解性樹脂コンクリートの力学特性に関する基礎的研究の先駆けとして、生分解 性樹脂コンクリートの強度変化とそのメカニズムを解明することを目的に、樹脂の種類、板厚、樹 脂混合率、分子量や埋設土の種類などを変化させ、三点曲げ試験や圧縮試験、微生物量測定などを 実施した.その結果、暴露期間の経過にともない生分解性樹脂コンクリートの曲げ強度と圧縮強度 が低下することが明らかとなった.強度低下傾向と微生物量には相関関係がなかったことや、空気 中では劣化しなかったことから、強度低下を引き起こす最大の原因は水であると結論付けた.ま た、強度試験後の破断面の詳細な観察より、樹脂部からの骨材の剥離が確認され、骨材と樹脂の付 着力の低下が強度低下に繋がることが示唆された.

さらに、分解性樹脂コンクリートの実用化にむけて、生分解性樹脂コンクリートパイプを作製 し、管軸方向圧縮試験、円周方向圧縮試験を実施した.その結果、生分解性樹脂コンクリートパイ プは、十分な初期強度を発現し、暴露後には、再開発に支障をきたさないほどの強度低下が見込め ることが明らかとなった.今後は、生分解性樹脂コンクリートパイプの実現により、非開削推進工法 への貢献が大いに期待できるものと推察され、社会貢献が大いに期待できる新しい土木材料である.

第5章では、生分解性樹脂コンクリートの劣化特性から、様々な外的劣化要因を包括できる劣化 モデル式を作成するために、ワイブル分布を用いた統計的劣化推定モデルを作成した.その結果、 生分解性樹脂コンクリートを実際の仮設資材へと適用する際に不可欠となる長期的な強度低下予測 や、強度保持期間を推定することが可能となり、実用化への大きな一歩であると考えられる.

第6章では、第2章から第5章の研究から得られた結果を総括した.