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NTSQP

China National Centre for Quality Supervision & Test of Plastic Products

Test Report

Sample clients*	Shenzhen Esun Industrial Co., Ltd
Sample producer*	Shenzhen Esun Industrial Co., Ltd
Sample name*	eSUN Biodegradable Polycaprolactone
Sample type*	PCL800C
Trade mark*	Polycaprolactone
Precatory code	2019E0622
Report number	NTSQP[2020]C0341
Date of Report	Jul. 30, 2020

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Sample Client *	Shenzhen Esun Industrial Co., Ltd			Telephone No.*	18696201688
Address*	465# Xiaowu Road, Xiaonan Economic Development Zone, Xiaogan, Hubei				
Sample Producer*	Shenzhen Esun Industrial Co., Ltd				
Name of Sample*	eSUN Biodegradable Polycaprolactone			Trade Mark*	Polycaprolactone
Type and specification*	PCL800C	Date of Production*	Aug. 1, 2019	Receiving Date	Nov. 25, 2019
Sampling Place	----	Quantity of the batch	----	Date of Sampling	----
Description of Sample	Samples were white resin and sheet made from the resin.				
Standard of Test	ASTM D 6400-12 "Standard Specification for Labeling of Plastics Designed to be Aerobically Composted in Municipal or Industrial Facilities" and EN 13432:2000 "Packaging-Requirements for packaging recoverable through composting and biodegradation-Test scheme and evaluation criteria for the final acceptance of packaging"				
Summary of test	7 items entrusted for test, 5 items passed and accepted, 2 items without conclusion.				
Inspection Conclusion	**03～07 items of the sample were qualified according to the Standard with ASTM D 6400-12 and EN 13432: 2000, 01～02 items of the sample were not conclusion for not specified in standard.**				
Remark	1.The client's objection, in case there is any, shall be submitted in the written form within 15days upon receipt of the test report. No acceptance shall be allowed when the objection is overdue. 2.Duplication of part of the test report shall not be allowed unless with the written approval of the Center. 3.The test report shall be invalid when it is incomplete. 4.The test report is responsible to the client only for the submitted sample(s) . 5. The content of the items with the symbol '*' is supplied by the Client and Center is not responsible to confirm it.				

Approved by: Shansheng Liu

Reviewed by: Yunxuan Weng

Compiled by: Yu Bai

No.	Item(s) Tested	Unit	Requirement		Test Result	Conclusion
01	Thickness					
	maximum thickness	mm	----		3.36	----
	minimum thickness	mm	----		3.26	----
	average thickness	mm	----		3.31	----
02	Material analysis	----	----		The main component of sample was PCL	----
03	Volatile solids content	%	>50		99.9	Passed
04	Maximum permissible heavy metal content		EN	USA		Passed
	Arsenic	mg/kg	5	20.5	0.214	
	Cadmium	mg/kg	0.5	19.5	0.177	
	Copper	mg/kg	50	750	<0.02	
	Lead	mg/kg	50	150	1.62	
	Mercury	mg/kg	0.5	8.5	<0.0003	
	Nickel	mg/kg	25	210	7.77	
	Selenium	mg/kg	0.75	50	0.380	
	Zinc	mg/kg	150	1400	<0.02	
	Chromium	mg/kg	50	-	0.704	
	Molybdenum	mg/kg	1	-	0.628	
	Fluorine	mg/kg	100	-	4.1	
05	The degree of biodegradation when compared to the positive control	%	≥90		98.0	Passed
06	Degree of disintegration	%	≥90		100.0	Passed
07	Ecotoxicity					
	Germination rate corresponding vlaues obtained with blank compost	%	≥90			Passed
	Summer barley (compost/soil, 1:1)				97.0	
	Summer barley (compost/soil, 1:3)				100.7	
	Cabbage (compost/soil, 1:1)				99.6	
	Cabbage (compost/soil, 1:3)				98.0	
	Biomass rate corresponding values with blank compost	%	≥90			Passed
	Summer barley (compost/soil, 1:1)				102.3	
	Summer barley (compost/soil, 1:3)				95.7	
	Cabbage (compost/soil, 1:1)				96.9	
	Cabbage (compost/soil, 1:3)				106.4	

Note: The detail of procedure of test item 01～07 was described in section 1～7 at annex of this report respectively.

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NTSQP
国家塑料制品质量监督检验中心（北京）
NTSQP

Section 1 Thickness

1.1 Guideline

GB/T 6672-2001 Plastics -- Film and sheeting -- Determination of thickness by mechanical scanning (IDT ISO 4593:1993)

1.2 Thickness measurement

Condition for testing was at (23±2)℃for at least 1h.

The specimens were cut in size of 10cm×10cm from sample sheet, then measured the thickness once of each specimens. Its average thickness was calculated after measure.

1.3 Result

As the test result, the measured maximum thickness of sheet was 3.36mm, minimum thickness was 3.26mm, the average thickness was 3.31mm.

Section 2 Components

2.1 Guideline

GB/T 6040-2002 General rules for infrared analysis

2.2 Measurement

Cut a little sample with scalpel and made test specimen by Diamond Anvil Cell (DAC), tested it by Fourier Transform Infrared Spectrometer.

2.3 IR spectrophotometer

The IR spectrum of test specimen and PCL were shown on fig.2-1 and fig.2-2 respectively.

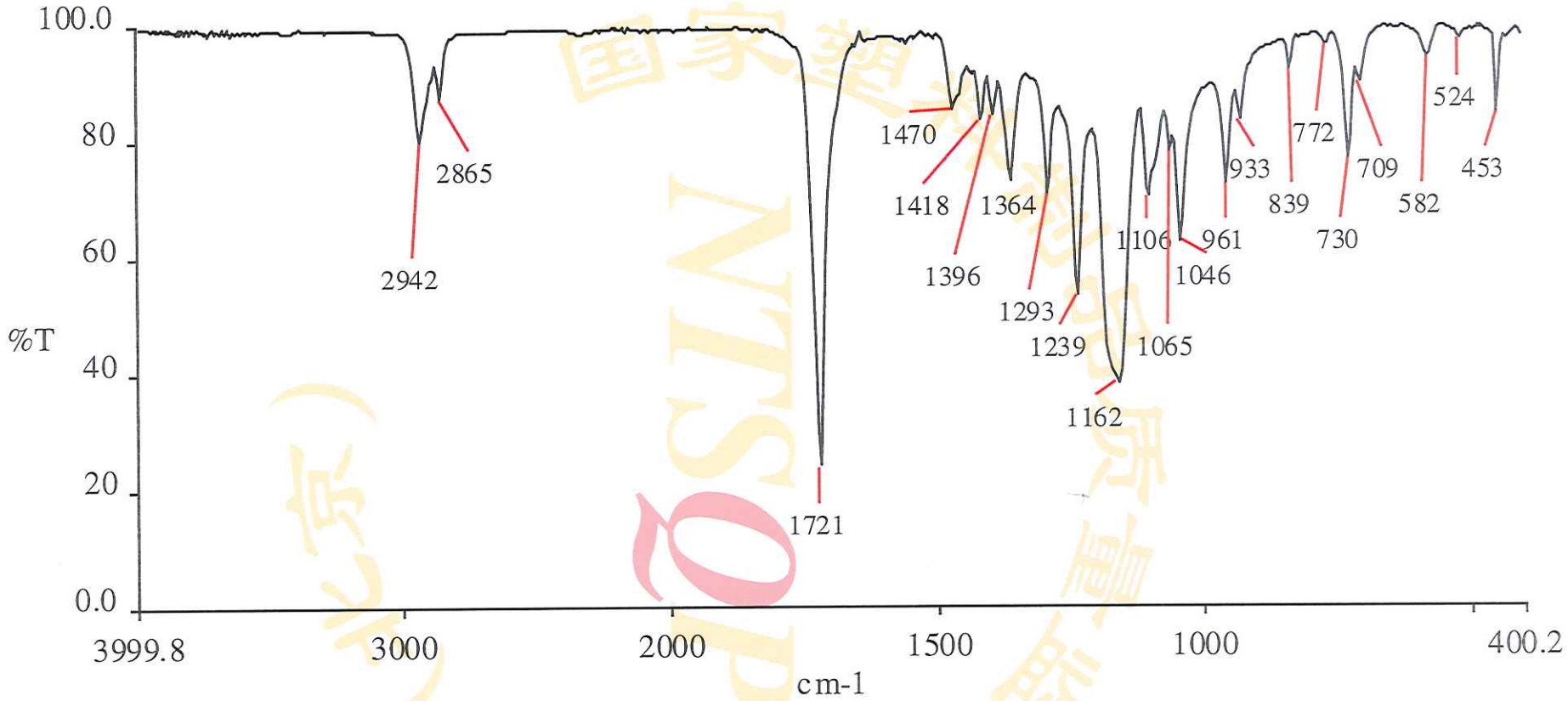

Fig.2-1 Infrared spectrum of sample

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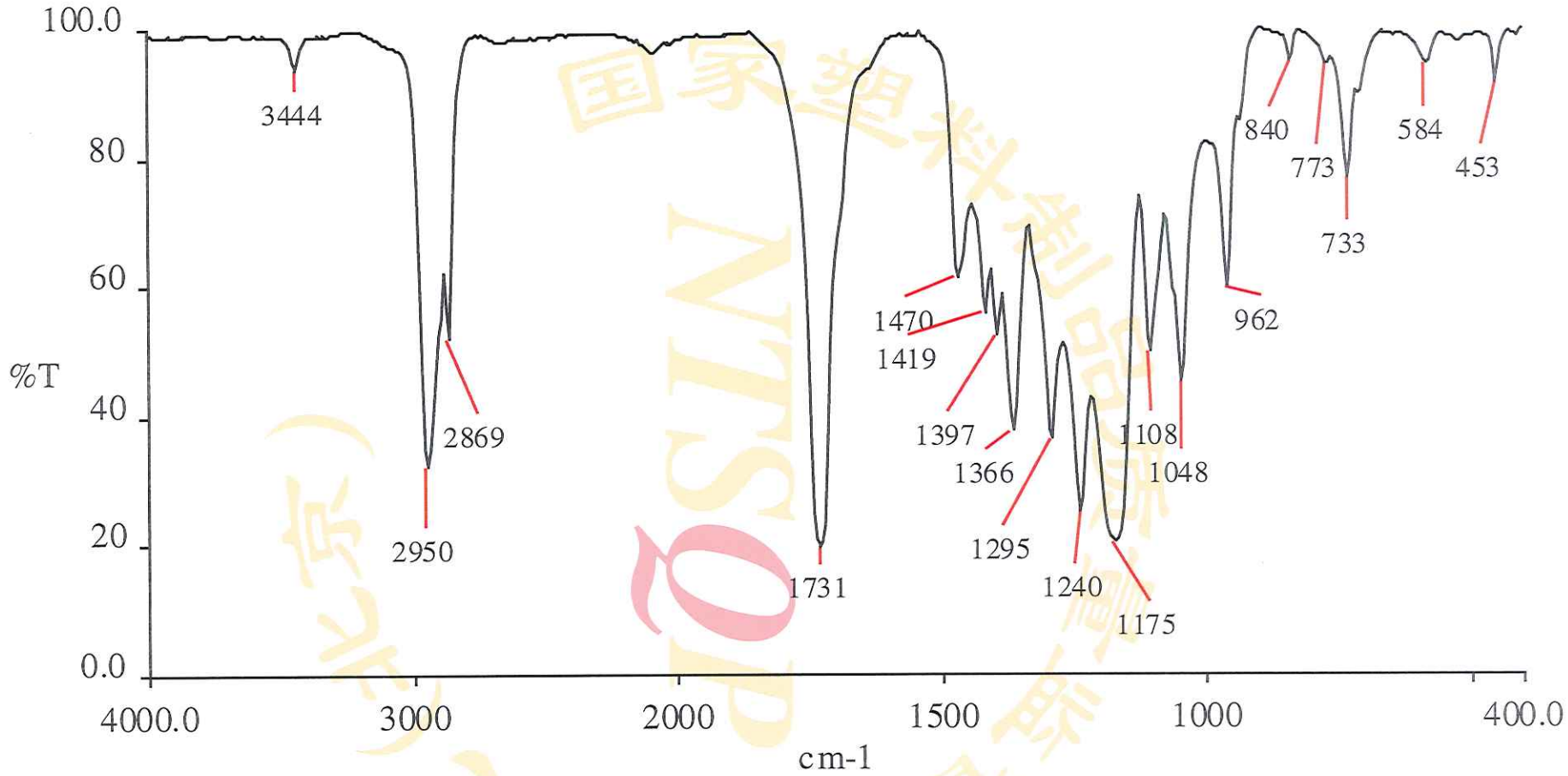

Fig.2-2 Infrared spectrum of PCL

2.4 Analysis

Compared spectrum in fig.2-1 and fig.2-2, it can be found that the main component of sample was PCL.

Section 3 Volatile solids content of sample

3.1 Guideline

GB/T 9345.1-2008 Plastics - Determination of ash - Part 1: General methods (IDT ISO 3451-1:1997)

3.2 Determination of volatile solids content of sample (VS)

Dried a crucible for 3h at 105℃, then took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_0);

Add about 3~10g sample (less than 2/3 of crucible volume) in crucible, put it in the drying oven and dried it for 3h at 105℃, took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_2);

Then moved it into the muffle furnace and incinerated it at 550℃ for 3h, took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_3);

Calculated the volatile solids content of sample as following,

$$\text{VS}\ (\%) = \frac{m_2 - m_3}{m_2 - m_0} \times 100$$

Repeated the test one time.

Determination result shall be expressed by the average value.

3.3 Result and discussion

The volatile solids content of sample was 99.9%, which was more than 50% as required in EN 13432.

Section 4 Heavy Metals

4.1 Guideline

ASTM D 6400-12 Standard Specification for Labeling of Plastics Designed to be Aerobically Composted in Municipal or Industrial Facilities

EN 13432:2000 Packaging-Requirements for packaging recoverable through composting and biodegradation-Test scheme and evaluation criteria for the final acceptance of packaging

4.2 Determination of heavy metals of the sample

Heavy metals were tested by atomic absorption spectroscopy after disposing the sample by high pressure microwave digestion.

To determine fluorine content, weighed about 0.5g sample, then pretreated by combustion and hydrolysis, and determined fluorine content with IC.

4.3 Result

The test result of heavy metals were listed on table 4-1.

Table 4-1 list of test result of heavy metal

Item	Unit	Test Result	EN13432	USA	Canada
Arsenic	mg/kg	0.214	5	20.5	19
Cadmium	mg/kg	0.177	0.5	19.5	5
Copper	mg/kg	<0.02	50	750	189
Lead	mg/kg	1.62	50	150	125
Mercury	mg/kg	<0.0003	0.5	8.5	1
Nickel	mg/kg	7.77	25	210	45
Selenium	mg/kg	0.380	0.75	50	4
Zinc	mg/kg	<0.02	150	1400	463
Chromium	mg/kg	0.704	50	-	265
Molybdenum	mg/kg	0.628	1	-	5
Fluorine	mg/kg	4.1	100	-	-
Cobalt	mg/kg	0.684	-	-	38

4.4 Result and discussion

It can be seen from table 4-1 that the content of each heavy metal of test sample was low than the requirement in EN 13432.

Section 5 Biodegradation testing under controlled aerobic composting

5.1 Introduction

1) Principle of test method

This testing determines the ultimate biodegradability of test material under conditions simulating an intensive aerobic composting process. The test material was mixed with the inoculum and introduced into a simulated composting vessel where it was intensively composted under optimum oxygen, temperature and moisture conditions for a test period not exceeding 6 months.

2) Guidelines

ISO 14855-1:2012 Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions – Method by analysis of evolved carbon dioxide – Part1: General method

ISO 13878-1998 Soil quality - Determination of total nitrogen content by dry combustion ("elemental analysis")

ISO 11465:1993 Soil quality - Determination of dry matter and water content on a mass basis - Gravimetric method

ISO 10390:1994 Soil quality -- Determination of pH

ISO 10304-2:1995 Water quality -- Determination of dissolved anions by liquid chromatography of ions -- Part 2: Determination of bromide, chloride, nitrate, nitrite, orthophosphate and sulfate in waste water

GB/T 9345.1-2008 Plastics - Determination of ash - Part 1: General methods (IDT ISO 3451-1:1997)

GB/T 7481-1987 Water quality -- Determination of ammonium -- Part 1: Manual spectrometric method (eqv ISO 7150-1:1984)

5.2 Materials

1) Test material

- Name: eSUN Biodegradable Polycaprolactone
- Description: white resin

2) Reference material

- Name: cellulose
- Description: white powder, 20μm

3) Inoculum

The compost was sampled from the composting factory in Beijing. The age of compost was 3 months (The grade after testing with Rottegrad method was Ⅴ).

5.3 Biodegradation testing

1) Apparatus

- Air flowmeter (precision，1.5%)
- Bio-test vessel (3L)
- IFR detector (precision，±2%)
- pH meter (precision，±0.05pH)
- Moisture meter (precision，±1%RH)
- Balance (precision，±0.1mg，measuring range 160g; precision，±0.1g，measuring range 610g)
- Drying oven (precision，±2℃)
- Element analyzer (precision，0.1%)
- Muffle furnace (precision，±10℃)
- Temp-consistent bath (precision，±0.5℃)

2) Schematic of apparatus

The schematic of testing apparatus was shown fig.5-1.

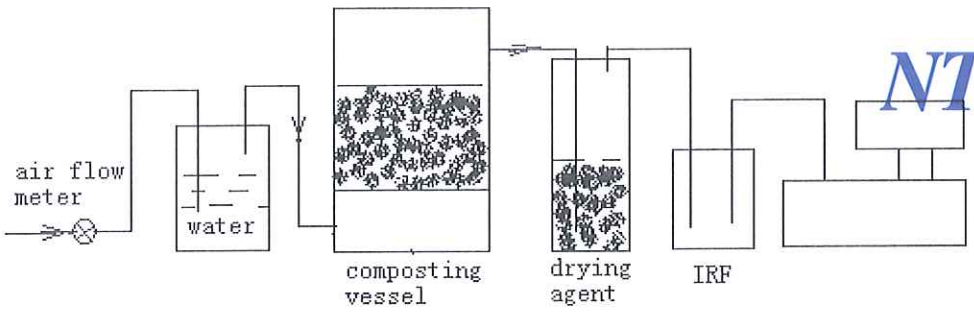

Fig. 5-1 Controlled composting apparatus schematic

3) Testing Methods

a. Determination of the total dry solids content (TDS) and volatile solids content (VS)

Dried a crucible for 3h at 105℃, then took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_0);

Added about 3~10g sample (less than 2/3 of crucible volume) in crucible, weighed it(record as m_1);

Then put it in the drying oven and dried it for 3h at 105℃, took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_2);

Then moved it into the muffle furnace and incinerated it at 550℃ for 3h, took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_3);

Calculated the total dry solids content of sample as following:

$$TDS\ (\%) = \frac{m_2 - m_0}{m_1 - m_0} \times 100$$

calculated the volatile solids content of sample as following,

$$\mathrm{VS}\ (\%) = \frac{m_2 - m_3}{m_2 - m_0} \times 100$$

Repeated the test one time.

Determination result shall be expressed by the average value.

b. Total Organic-carbon and nitrogen content

The total organic carbon(TOC) and nitrogen content were measured by elemental analyzer. Took about 2~5mg sample into the test cell of elemental analyzer and determined it under 1150℃ with CHNO mode of procedure.

c. Theoretical evolved carbon dioxide($ThCO_2$) of test sample

Calculated the theoretical evolved carbon dioxide: $ThCO_2 = TOC \times 44/12$. (1mol C≈1mol CO_2)

44, molar mass of CO_2;

12, molar mass of Carbon(C).

d. pH value

Determined the pH value of compost using following steps. Prepared a mixture of 1 part of compost with 5 parts of deionized water which means that 20g±0.1g compost was put into a 200ml beaker, added 100g±0.1g of deionized water, shook it, measured pH immediately with pH meter.

e. Determination of carbon dioxide evolved

Turned on infrared analyzer to measure the evolved carbon dioxide of each vessel. Evolved carbon dioxide was determined by IFR detector which sent the data to the computer and the computer recorded the data in a database with the test program.

4) Test procedure

a) Prepared three containers used as the blank test vessels. To each container was added 600g±0.1g (dry weight) compost. Added water to the mixture to achieve humidity of about 50%.

b) Prepared three containers, used as reference material testing vessels. To each container was added 600g±0.1g (dry weight) of compost and 100g±0.1g (dry weight) of cellulose. Fully mixed the mixture and add water to the mixture to achieve humidity of about 50%.

c) Prepared three containers, used as test materials vessels. To each container was added 600g±0.1g (dry weight) of compost and 100g±0.1g (dry weight) samples. Fully mixed the mixture, added water to it to achieve

humidity of about 50%.

d) Sealed the vessels and put them in a water bath with constant temperature 58℃±2℃. Supplied each container with compressed 200ml/min±3ml/min air flow.

e) Ran the computer program of "biodegradation testing control system". Turned on infrared analyzer to measure the evolved carbon dioxide of each vessel.

f) Measured the oxygen concentration in the exhaust air in twice daily.

g) Shake the composting vessels weekly to prevent extensive channeling and to ensure uniform attack of the microorganisms on the test material. It was helpful in ensuring an even distribution of moisture.

i) Calculated the volume of carbon dioxide released from the every reactor vessel each day.

j) Calculated the evolved carbon dioxide of vessels of test materials and reference materials after subtracting that of vessel of blank testing. From the cumulative amounts of carbon dioxide released, calculated the percentage biodegradation D_t(%) of the test material or reference material for each measurement interval using following equation:

$$D_t = \frac{(CO_2)_T - (CO_2)_B}{ThCO_2} \times 100$$

Where,

$(CO_2)_T$ was the cumulative amount of carbon dioxide evolved in each composting vessel containing test material, in grams per vessel;

$(CO_2)_B$ was the mean cumulative amount of carbon dioxide evolved in the blank vessels, in grams per vessel;

$ThCO_2$ was the theoretical amount of carbon dioxide which can be produced by the test material, in grams per vessel.

k) Period of the test

Incubated the composting vessels for a period not exceeding 6 months at a constant temperature of 58℃±2℃ which was representative of full-scale composting.

5.4 Result and discussion

1) The basic properties of test sample and reference material

The granule of sample was used as test sample and the powder of cellulose was used as reference materials respectively in this testing. The TOC, total dry solids and volatile solids of the test sample and reference materials were tested and $ThCO_2$ was calculated with TOC. The results were listed on table 5-1.

Table 5-1 basic properties of samples

Samples	Dry solids (%)	Moisture (%)	TOC of dry sample (%)	$ThCO_2$ (CO_2g/100g dry sample)
Test sample	99.45	0.55	57.78	211.86
Reference material	99.76	0.24	44.51	163.20

It can be seen from table 5-1, that the dry solids of test sample was 99.45% and the TOC of dry sample was 57.78% which mean that 50g total dry solids of sample contained 28.89g TOC. As required in ISO 14855-1, a minimum of 50g of total dry solids of test material should contain 20g of TOC. It was said that the test sample had sufficient organic carbon to yield carbon dioxide in an amount suitable for the determination.

2) Basic property of inoculum

Used the compost sampled from compost factory and determined the TDS, VS of inoculum. The result was listed on table 5-2.

Table 5-2 basic property of inoculums

dry solids(TDS), %	moisture, %	volatile solids(VS),%	ratio of carbon and nitrogen(C/N)
74.8	25.2	25.8	19.9

It can be seen from the table 5-2, that the volatile solids of inoculum was 25.8% of the dry solids. The dry solids content of inoculum was 74.8%. Adjust the TDS to 50%～55% by added water before the compost was used.

3) Actual material added into vessel

The actual material added to each vessel of testing group was listed on table 5-3.

Table 5-3 actual material added to each vessel of each testing group

Vessel	Inoculums		Testing sample or reference material		Ratio of carbon to nitrogen(C/N)
	wet weight(g)	dry weight(g)	wet weight (g)	dry weight(g)	
Sample vessel	802.5	600.0	100.6	100.0	25.3
Blank vessel	802.5	600.0	——	——	19.9
Reference material vessel	802.5	600.0	100.2	100.0	23.3

Note: the value of C/N of each vessel was calculated based on the drying weight of inoculum, test sample and reference material respectively.

4) pH changing

Determined the pH value of inoculum using following steps. Prepare a mixture of 1 part of inoculum with 5 parts of deionized water which means that 20g±0.1g compost was put into a 200ml beaker, add 100g±0.1g of deionized water, shake it, measure pH immediately with pH meter.

The results of pH value of inoculum of each test vessel at different period were listed on table 5-4.

Table 5-4 pH value of each vessel

Day	Blank container			Reference container			Sample container		
	1#	2#	3#	1#	2#	3#	1#	2#	3#
0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0
7	7.0	7.1	7.1	7.1	7.1	7.2	7.1	7.0	7.1
14	7.1	7.2	7.2	7.2	7.1	7.2	7.0	7.1	7.2
21	7.1	7.2	7.3	7.3	7.2	7.3	7.2	7.2	7.2
28	7.2	7.3	7.3	7.4	7.2	7.4	7.1	7.2	7.3
35	7.3	7.4	7.4	7.4	7.4	7.5	7.2	7.3	7.2
42	7.4	7.4	7.4	7.5	7.5	7.5	7.3	7.3	7.3
49	7.4	7.5	7.5	7.6	7.6	7.6	7.3	7.4	7.5
56	7.5	7.5	7.6	7.6	7.7	7.7	7.4	7.4	7.6
63	7.6	7.6	7.8	7.7	7.8	7.6	7.5	7.6	7.5
70	7.6	7.7	7.9	7.8	7.8	7.8	7.4	7.7	7.6
77	7.7	7.7	7.9	7.8	7.8	7.9	7.6	7.9	7.6
84	7.8	7.8	8.0	7.9	7.7	8.0	7.8	7.9	7.7
91	7.8	7.9	8.2	7.9	7.8	8.1	7.7	8.0	7.8
98	7.9	8.0	8.1	8.0	7.9	8.2	7.9	8.0	7.8
105	7.9	8.1	8.2	8.1	8.0	8.2	7.9	8.1	7.9
112	8.0	8.1	8.3	8.2	8.2	8.3	8.0	8.0	7.9
119	8.1	8.2	8.4	8.3	8.2	8.4	8.0	8.2	8.1
126	8.1	8.3	8.5	8.3	8.3	8.3	8.2	8.2	8.0
133	8.2	8.4	8.6	8.2	8.2	8.4	8.3	8.3	8.1
140	8.2	8.5	8.7	8.3	8.3	8.4	8.4	8.5	8.2
147	8.3	8.5	8.6	8.4	8.3	8.5	8.4	8.4	8.3
154	8.4	8.6	8.7	8.4	8.2	8.6	8.6	8.5	8.5
161	8.4	8.6	8.7	8.5	8.3	8.6	8.6	8.6	8.4
168	8.5	8.7	8.6	8.5	8.4	8.7	8.7	8.5	8.5
175	8.4	8.6	8.7	8.6	8.4	8.7	8.7	8.6	8.6
180	8.5	8.7	8.6	8.5	8.5	8.6	8.7	8.7	8.6

It can be seen from table 5-4, the pH value of each vessel was 7.0 at the beginning of test. The pH value of each vessel was increased with the testing going on and the highest value was 8.7 until the test was finished. All of pH value of each vessel was lower than 9.0 which was corresponded with the requirement of standard.

The result of visual observation of the inoculums was described as following:

At beginning of test: the sample was white granules, which was pulverized into powder and scraps.

Determined TDS and VS of sample scraps and compost respectively. Later check the results with standard requirements (VS>50% in dry solid as sample, VS<30% in dry solid as compost). Accurately weighed out 600g of inoculum in dry weight, and 100g of sample in dry solids. Added water to adjust the moisture of mixture to about 50%, then fully mixed it again.

At 1st week: the color of compost was brown and sample scraps were white. They were distinguished clearly. The odor of mixture was not olid at this time. Mildew or fungus were not found easily. The structure was loose, no block and clump.

At 2nd week: the amount of sample scraps decreased obviously. Most of the scraps turned black or the same color with compost. They were distinguished not easily. Other scraps turned bronze. There were white and green mildew spots appeared around the scraps. the odor of compost was olid clearly.

At 3rd week: the amount of mildew spots increased than last week. Most of them were white and green, others were yellowy. Mildew spots could be seen on the surface or inner the compost. Sample scraps were found not easily at this time. The fine sample scraps were dark brown which could be found after careful search.

At 4th week: sample scraps were found difficult in the mixture. The compost mixture was evenly deep puce, no block and clump. The odor of mixture was olid clearly and could be smelt immediately when lid opened. There was no significant change in the number and morphology of mildew spots.

At 5th week: the odor of mixture decreased sharply, no such olid smell than last week. It was mature and sour smell. The amount of mildew spots decreased. Some of the spots deepened to puce. The structure of compost mixture was loose, no block and clump.

At 6th week: sample scraps almost disappeared in compost, and could not be found after careful search. The amount of mildew spots decreased. Most of them were yellow, green and tan. The odor of mixture was still slight olid and sour smell.

At 7th week: the amount of mildew and fungus decreased continuously. Most of them were dark gray and puce. The smell of mixture was slight olid and sour. The color of compost mixture was dark black. The structure was loose, no block and clump.

After 8th to 10th week: the appearance of compost mixture was not changed obviously, as the characteristics of sense was not changed clearly. The odor was light mature, no olid and sour smell. The color was dark black. No mildew or fungus could be seen.

11th week to the ending: the stability of appearance and sensory characteristics of compost mixture continued, no significant change. No visible mildew or fungus at this time. The odor was not olid, but light moldy and moist yet.

5) The result of carbon dioxide evolved and percentage biodegradation

The results of carbon dioxide evolved of each vessel, it's average, and the average evolved carbon dioxide subtracting that of blank, and degree of biodegradation were listed in table 5-5. Plot the average of cumulative amount of carbon dioxide evolved for three composting vessels containing blank, test material and reference material as a function of time, see fig.5-2. Plot a biodegradation curve (percentage biodegradation as a function of time) for the test material and the reference, see fig.5-3.

Table 5-5 the evolved of CO_2 and degree of biodegradation

Day	Blank container				Reference material container (cellulose)				Sample container (2019E0622)				Average evolved CO_2 subtracting that of blank, g		Degree of biodegradation (Dt),%	
	1#	2#	3#	average	1#	2#	3#	average	1#	2#	3#	average	Cellulose	sample	Cellulose	sample
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1	2.70	3.08	2.56	2.78	3.28	3.41	3.39	3.36	3.06	3.72	3.00	3.26	0.58	0.48	0.36	0.23
2	4.37	4.73	3.98	4.36	6.33	6.83	7.39	6.85	5.97	6.75	6.63	6.45	2.49	2.09	1.53	0.99
3	6.00	6.44	6.01	6.15	10.70	10.40	11.12	10.74	9.26	9.63	10.75	9.88	4.59	3.73	2.81	1.76
4	8.59	8.04	6.95	7.86	13.94	14.20	15.60	14.58	12.04	12.26	14.97	13.09	6.72	5.23	4.12	2.47
5	9.91	9.01	8.14	9.02	18.54	17.69	18.91	18.38	15.73	15.60	18.86	16.73	9.36	7.71	5.74	3.64

Day	Blank container				Reference material container (cellulose)				Sample container (2019E0622)				Average evolved CO_2 subtracting that of blank, g		Degree of biodegradation (Dt),%	
	1#	2#	3#	average	1#	2#	3#	average	1#	2#	3#	average	Cellulose	sample	Cellulose	sample
6	10.85	10.80	11.38	11.01	22.15	21.25	23.23	22.21	19.51	18.53	21.81	19.95	11.20	8.94	6.86	4.22
7	13.69	11.76	12.83	12.76	25.33	23.90	25.29	24.84	20.61	20.61	25.65	22.29	12.08	9.53	7.40	4.50
8	14.56	13.59	13.64	13.93	28.54	28.95	33.17	30.22	24.11	24.89	29.36	26.12	16.29	12.19	9.98	5.75
9	16.80	14.77	13.67	15.08	32.87	33.60	38.44	34.97	27.84	28.36	33.89	30.03	19.89	14.95	12.19	7.06
10	18.06	15.64	14.66	16.12	36.68	38.00	43.58	39.42	31.63	31.54	37.54	33.57	23.30	17.45	14.28	8.24
11	19.20	16.69	16.07	17.32	40.38	42.40	49.40	44.06	33.86	35.42	40.76	36.68	26.74	19.36	16.38	9.14
12	20.83	17.86	17.62	18.77	45.46	47.03	53.46	48.65	37.58	38.84	44.33	40.25	29.88	21.48	18.31	10.14
13	22.13	19.17	18.91	20.07	49.17	51.38	59.14	53.23	40.94	43.09	47.19	43.74	33.16	23.67	20.32	11.17
14	24.20	20.78	20.33	21.77	55.15	56.02	62.32	57.83	43.61	46.21	50.97	46.93	36.06	25.16	22.10	11.88
15	25.94	21.99	21.82	23.25	59.37	60.32	67.54	62.41	46.52	49.94	53.99	50.15	39.16	26.90	24.00	12.70
16	27.34	23.43	23.51	24.76	65.52	64.93	70.61	67.02	49.51	53.07	58.82	53.80	42.26	29.04	25.89	13.71
17	28.87	24.54	24.56	25.99	71.46	69.46	73.88	71.60	51.99	56.10	62.88	56.99	45.61	31.00	27.95	14.63
18	29.78	25.33	25.14	26.75	77.29	74.65	78.28	76.74	54.74	59.37	66.58	60.23	49.99	33.48	30.63	15.80
19	30.96	26.52	26.40	27.96	83.22	77.35	78.56	79.71	59.53	64.93	69.91	64.79	51.75	36.83	31.71	17.38
20	31.66	27.20	27.24	28.70	87.85	81.30	83.15	84.10	62.21	68.11	74.31	68.21	55.40	39.51	33.95	18.65
21	32.59	27.91	28.18	29.56	93.42	85.51	85.52	88.15	65.52	73.24	78.71	72.49	58.59	42.93	35.90	20.26
22	33.63	28.78	28.94	30.45	97.74	89.45	89.47	92.22	69.55	76.48	83.02	76.35	61.77	45.90	37.85	21.67
23	34.67	29.62	29.91	31.40	101.70	93.24	93.84	96.26	72.65	80.34	87.55	80.18	64.86	48.78	39.74	23.02
24	35.60	30.23	30.68	32.17	104.76	97.46	98.62	100.28	76.30	84.01	92.47	84.26	68.11	52.09	41.73	24.59
25	36.34	30.87	31.73	32.98	108.19	101.69	103.17	104.35	79.97	86.85	96.49	87.77	71.37	54.79	43.73	25.86
26	37.49	31.83	32.56	33.96	111.80	105.37	108.03	108.40	83.44	90.07	99.88	91.13	74.44	57.17	45.61	26.98
27	38.35	32.79	33.53	34.89	115.72	110.09	114.15	113.32	86.43	94.46	103.21	94.70	78.43	59.81	48.06	28.23
28	39.67	33.87	34.43	35.99	120.98	114.23	118.58	117.93	89.90	97.67	106.91	98.16	81.94	62.17	50.21	29.34
29	40.70	34.67	35.39	36.92	124.70	119.41	125.07	123.06	91.87	100.69	109.54	100.70	86.14	63.78	52.78	30.10
30	41.42	35.22	35.98	37.54	128.10	123.75	131.64	127.83	95.42	105.07	114.63	105.04	90.29	67.50	55.32	31.86
31	42.00	35.80	36.74	38.18	136.49	131.72	141.56	136.59	100.31	109.62	120.91	110.28	98.41	72.10	60.30	34.03
32	42.80	36.55	37.86	39.07	140.18	135.07	145.71	140.32	102.70	112.10	123.72	112.84	101.25	73.77	62.04	34.82
33	43.60	37.19	38.46	39.75	144.77	139.57	148.47	144.27	105.61	115.21	128.56	116.46	104.52	76.71	64.04	36.21
34	44.62	37.95	39.17	40.58	146.88	141.48	153.09	147.15	109.20	119.31	132.39	120.30	106.57	79.72	65.30	37.63
35	45.53	38.67	39.73	41.31	150.23	143.55	154.45	149.41	112.07	123.40	135.42	123.63	108.10	82.32	66.24	38.86
36	46.11	39.23	40.24	41.86	153.41	145.77	156.01	151.73	116.28	126.40	138.95	127.21	109.87	85.35	67.32	40.29
37	46.61	39.68	40.88	42.39	155.12	147.77	158.15	153.68	119.91	131.05	144.62	131.86	111.29	89.47	68.19	42.23
38	47.18	40.11	41.38	42.89	158.95	151.13	161.82	157.30	123.40	134.76	147.83	135.33	114.41	92.44	70.10	43.63
39	47.67	40.56	42.06	43.43	160.63	152.64	163.28	158.85	127.75	138.86	150.57	139.06	115.42	95.63	70.72	45.14
40	48.31	41.09	42.54	43.98	163.01	156.02	168.80	162.61	131.63	142.14	152.44	142.07	118.63	98.09	72.69	46.30
41	48.76	41.47	43.12	44.45	166.13	159.05	171.86	165.68	134.29	145.03	157.36	145.56	121.23	101.11	74.28	47.72
42	49.15	41.86	43.63	44.88	169.49	163.27	176.70	169.82	137.31	147.55	161.30	148.72	124.94	103.84	76.56	49.01
43	49.68	42.45	44.25	45.46	173.14	165.45	177.92	172.17	140.66	150.19	164.13	151.66	126.71	106.20	77.64	50.13
44	50.23	42.87	44.72	45.94	174.84	166.55	178.33	173.24	144.97	153.80	168.39	155.72	127.30	109.78	78.00	51.82
45	50.79	43.29	45.24	46.44	177.59	167.83	178.59	174.67	148.53	157.69	170.51	158.91	128.23	112.47	78.57	53.09

Day	Blank container				Reference material container (cellulose)				Sample container (2019E0622)				Average evolved CO_2 subtracting that of blank, g		Degree of biodegradation (Dt),%	
	1#	2#	3#	average	1#	2#	3#	average	1#	2#	3#	average	Cellulose	sample	Cellulose	sample
46	51.57	43.95	45.90	47.14	178.59	168.73	179.36	175.56	151.61	161.95	174.48	162.68	128.42	115.54	78.69	54.54
47	51.89	44.27	46.28	47.48	179.13	169.40	180.07	176.20	155.02	163.96	178.78	165.92	128.72	118.44	78.87	55.90
48	52.42	44.69	46.68	47.93	181.48	170.65	180.46	177.53	159.60	165.01	180.59	168.40	129.60	120.47	79.41	56.86
49	52.94	45.15	47.05	48.38	183.84	173.31	183.06	180.07	163.31	168.24	186.52	172.69	131.69	124.31	80.69	58.68
50	53.64	45.68	47.56	48.96	185.64	175.11	184.68	181.81	167.28	171.33	189.75	176.12	132.85	127.16	81.40	60.02
51	54.19	46.11	48.17	49.49	188.87	177.94	187.05	184.62	170.50	174.89	193.38	179.59	135.13	130.10	82.80	61.41
52	54.74	46.60	48.81	50.05	190.24	178.83	187.76	185.61	173.85	176.75	195.31	181.97	135.56	131.92	83.06	62.27
53	55.67	47.39	49.40	50.82	190.95	180.42	190.50	187.29	177.24	178.91	196.84	184.33	136.47	133.51	83.62	63.02
54	56.36	47.94	49.96	51.42	192.76	182.27	192.45	189.16	180.37	182.27	198.99	187.21	137.74	135.79	84.40	64.09
55	56.77	48.37	50.44	51.86	194.94	183.76	193.46	190.72	182.15	183.52	201.39	189.02	138.86	137.16	85.09	64.74
56	57.34	48.85	51.01	52.40	196.46	185.32	195.39	192.39	185.18	185.24	202.46	190.96	139.99	138.56	85.78	65.40
57	58.17	49.56	51.96	53.23	197.81	186.50	196.73	193.68	191.73	186.31	203.93	193.99	140.45	140.76	86.06	66.44
58	59.03	50.19	52.66	53.96	198.71	187.46	197.78	194.65	193.32	187.87	204.65	195.28	140.69	141.32	86.21	66.70
59	59.81	50.79	53.53	54.71	200.39	189.10	199.17	196.22	195.08	190.94	206.90	197.64	141.51	142.93	86.71	67.46
60	60.93	51.81	54.72	55.82	201.27	189.84	200.04	197.05	196.54	193.00	209.17	199.57	141.23	143.75	86.54	67.85
61	61.81	52.54	55.66	56.67	202.15	190.69	201.07	197.97	198.58	194.73	210.56	201.29	141.30	144.62	86.58	68.26
62	62.69	53.24	56.48	57.47	203.75	192.23	202.52	199.50	200.36	196.24	212.82	203.14	142.03	145.67	87.03	68.76
63	63.37	53.91	57.11	58.13	204.90	193.47	203.73	200.70	202.61	198.50	214.67	205.26	142.57	147.13	87.36	69.45
64	64.26	54.71	58.00	58.99	205.97	194.37	204.76	201.70	204.61	200.66	215.97	207.08	142.71	148.09	87.44	69.90
65	65.34	55.45	58.85	59.88	206.34	194.77	205.16	202.09	206.47	202.81	217.27	208.85	142.21	148.97	87.14	70.32
66	65.99	55.96	59.28	60.41	206.95	195.34	205.87	202.72	208.23	205.08	218.70	210.67	142.31	150.26	87.20	70.92
67	66.41	56.38	59.82	60.87	207.39	195.79	206.27	203.15	209.85	206.39	220.12	212.12	142.28	151.25	87.18	71.39
68	66.79	56.65	60.16	61.20	208.40	196.82	207.23	204.15	211.56	208.60	221.51	213.89	142.95	152.69	87.59	72.07
69	67.15	56.93	60.51	61.53	208.70	197.15	207.62	204.49	212.92	210.15	222.32	215.13	142.96	153.60	87.60	72.50
70	67.45	57.17	60.69	61.77	209.40	197.80	208.31	205.17	214.10	211.46	223.97	216.51	143.40	154.74	87.87	73.04
71	67.80	57.45	61.08	62.11	210.14	198.50	209.03	205.89	215.16	212.68	225.05	217.63	143.78	155.52	88.10	73.41
72	67.95	57.60	61.23	62.26	210.80	199.14	209.65	206.53	216.88	214.46	226.38	219.24	144.27	156.98	88.40	74.10
73	68.52	58.03	61.67	62.74	211.38	199.69	210.23	207.10	218.76	216.20	227.71	220.89	144.36	158.15	88.46	74.65
74	68.83	58.27	61.90	63.00	211.95	200.21	210.88	207.68	220.63	218.00	229.41	222.68	144.68	159.68	88.65	75.37
75	69.10	58.48	62.11	63.23	212.25	200.48	211.21	207.98	222.32	219.74	231.20	224.42	144.75	161.19	88.69	76.08
76	69.14	58.52	62.15	63.27	212.66	200.91	211.72	208.43	224.56	221.92	234.49	226.99	145.16	163.72	88.95	77.28
77	69.30	58.66	62.30	63.42	212.95	201.20	212.07	208.74	226.30	223.77	235.49	228.52	145.32	165.10	89.04	77.93
78	69.56	58.90	62.52	63.66	213.33	201.58	212.51	209.14	228.41	226.29	237.13	230.61	145.48	166.95	89.14	78.80
79	69.63	58.98	62.61	63.74	214.23	202.54	213.41	210.06	230.43	228.65	239.14	232.74	146.32	169.00	89.66	79.77
80	69.92	59.23	62.82	63.99	214.74	203.04	214.02	210.60	231.65	229.72	240.51	233.96	146.61	169.97	89.83	80.23
81	70.04	59.32	62.97	64.11	214.95	203.25	214.26	210.82	232.60	230.52	241.52	234.88	146.71	170.77	89.90	80.61
82	70.17	59.44	63.11	64.24	215.43	203.71	214.73	211.29	234.02	231.68	242.78	236.16	147.05	171.92	90.10	81.15
83	70.36	59.61	63.35	64.44	216.24	204.44	215.47	212.05	235.17	232.80	244.29	237.42	147.61	172.98	90.45	81.65
84	70.43	59.67	63.43	64.51	216.69	204.85	215.99	212.51	236.40	233.98	245.78	238.72	148.00	174.21	90.69	82.23
85	70.49	59.74	63.51	64.58	217.22	205.34	216.59	213.05	237.65	235.21	246.60	239.82	148.47	175.24	90.97	82.72

Day	Blank container				Reference material container (cellulose)				Sample container (2019E0622)				Average evolved CO_2 subtracting that of blank, g		Degree of biodegradation (Dt),%	
	1#	2#	3#	average	1#	2#	3#	average	1#	2#	3#	average	Cellulose	sample	Cellulose	sample
86	70.55	59.79	63.55	64.63	217.63	205.74	217.01	213.46	238.88	236.33	247.43	240.88	148.83	176.25	91.19	83.19
87	70.60	59.83	63.61	64.68	217.75	205.86	217.13	213.58	239.83	237.34	248.62	241.93	148.90	177.25	91.24	83.66
88	70.67	59.87	63.68	64.74	217.99	206.11	217.39	213.83	241.89	238.92	250.77	243.86	149.09	179.12	91.35	84.55
89	70.72	59.91	63.74	64.79	218.17	206.29	217.57	214.01	244.25	240.83	252.95	246.01	149.22	181.22	91.43	85.54
90	70.74	59.93	63.76	64.81	218.31	206.42	217.75	214.16	245.66	242.06	254.84	247.52	149.35	182.71	91.51	86.24
91	70.76	59.95	63.78	64.83	218.50	206.59	217.96	214.35	247.47	244.49	256.78	249.58	149.52	184.75	91.62	87.20
92	70.79	59.97	63.82	64.86	218.78	206.85	218.26	214.63	249.50	246.43	259.38	251.77	149.77	186.91	91.77	88.22
93	70.82	60.00	63.88	64.90	219.05	207.13	218.61	214.93	250.60	247.34	260.88	252.94	150.03	188.04	91.93	88.76
94	70.87	60.05	63.93	64.95	219.21	207.28	218.78	215.09	252.03	248.96	261.88	254.29	150.14	189.34	92.00	89.37
95	70.90	60.08	63.96	64.98	219.55	207.63	219.17	215.45	253.22	249.99	262.90	255.37	150.47	190.39	92.20	89.87
96	70.91	60.09	64.00	65.00	219.95	208.03	219.57	215.85	254.11	250.71	264.02	256.28	150.85	191.28	92.43	90.29
97	70.95	60.12	64.05	65.04	220.31	208.39	219.93	216.21	254.75	251.27	264.56	256.86	151.17	191.82	92.63	90.54
98	70.98	60.14	64.09	65.07	220.52	208.60	220.14	216.42	255.52	252.26	265.20	257.66	151.35	192.59	92.74	90.90
99	71.02	60.17	64.14	65.11	220.76	208.81	220.38	216.65	257.23	254.09	266.49	259.27	151.54	194.16	92.86	91.65
100	71.04	60.19	64.19	65.14	220.82	208.86	220.45	216.71	258.04	255.03	267.53	260.20	151.57	195.06	92.87	92.07
101	71.07	60.21	64.23	65.17	220.93	208.97	220.62	216.84	258.98	255.81	268.60	261.13	151.67	195.96	92.94	92.50
102	71.10	60.23	64.27	65.20	221.18	209.21	220.94	217.11	259.77	256.80	269.22	261.93	151.91	196.73	93.08	92.86
103	71.14	60.26	64.32	65.24	221.33	209.35	221.13	217.27	260.44	257.51	270.00	262.65	152.03	197.41	93.16	93.18
104	71.17	60.28	64.36	65.27	221.47	209.50	221.29	217.42	261.02	258.19	270.60	263.27	152.15	198.00	93.23	93.46
105	71.19	60.31	64.40	65.30	221.56	209.59	221.38	217.51	261.10	258.26	270.69	263.35	152.21	198.05	93.27	93.48
106	71.23	60.34	64.45	65.34	221.65	209.68	221.47	217.60	261.55	258.74	271.02	263.77	152.26	198.43	93.30	93.66
107	71.26	60.37	64.48	65.37	221.76	209.79	221.58	217.71	261.69	258.91	271.19	263.93	152.34	198.56	93.35	93.72
108	71.29	60.40	64.54	65.41	221.89	209.92	221.74	217.85	261.93	259.15	271.40	264.16	152.44	198.75	93.41	93.81
109	71.32	60.42	64.58	65.44	221.97	209.99	221.83	217.93	262.12	259.36	271.57	264.35	152.49	198.91	93.44	93.89
110	71.36	60.45	64.63	65.48	222.20	210.21	222.13	218.18	262.42	259.61	271.92	264.65	152.70	199.17	93.57	94.01
111	71.39	60.47	64.67	65.51	222.41	210.41	222.38	218.40	262.60	259.82	272.04	264.82	152.89	199.31	93.68	94.08
112	71.42	60.49	64.71	65.54	222.56	210.56	222.53	218.55	262.79	260.02	272.19	265.00	153.01	199.46	93.76	94.15
113	71.46	60.52	64.76	65.58	222.70	210.67	222.67	218.68	262.94	260.19	272.32	265.15	153.10	199.57	93.81	94.20
114	71.49	60.54	64.80	65.61	222.78	210.74	222.76	218.76	263.03	260.28	272.38	265.23	153.15	199.62	93.84	94.22
115	71.51	60.57	64.84	65.64	222.92	210.88	222.90	218.90	263.08	260.34	272.45	265.29	153.26	199.65	93.91	94.24
116	71.54	60.59	64.88	65.67	223.07	211.02	223.06	219.05	263.12	260.38	272.49	265.33	153.38	199.66	93.98	94.24
117	71.58	60.62	64.93	65.71	223.26	211.20	223.32	219.26	263.15	260.41	272.52	265.36	153.55	199.65	94.09	94.24
118	71.60	60.65	64.97	65.74	223.58	211.51	223.68	219.59	263.20	260.45	272.58	265.41	153.85	199.67	94.27	94.25
119	71.63	60.67	65.01	65.77	223.78	211.70	223.92	219.80	263.25	260.50	272.66	265.47	154.03	199.70	94.38	94.26
120	71.66	60.70	65.07	65.81	224.01	211.94	224.17	220.04	263.32	260.56	272.74	265.54	154.23	199.73	94.50	94.27
121	71.68	60.72	65.12	65.84	224.20	212.14	224.38	220.24	263.33	260.58	272.80	265.57	154.40	199.73	94.61	94.27
122	71.70	60.74	65.17	65.87	224.36	212.30	224.57	220.41	263.40	260.64	272.88	265.64	154.54	199.77	94.69	94.29
123	71.73	60.76	65.21	65.90	224.54	212.49	224.77	220.60	263.46	260.70	272.97	265.71	154.70	199.81	94.79	94.31
124	71.77	60.79	65.26	65.94	224.72	212.64	224.95	220.77	263.54	260.77	273.03	265.78	154.83	199.84	94.87	94.33
125	71.81	60.82	65.31	65.98	225.03	212.94	225.24	221.07	263.64	260.87	273.10	265.87	155.09	199.89	95.03	94.35

Day	Blank container				Reference material container (cellulose)				Sample container (2019E0622)				Average evolved CO_2 subtracting that of blank, g		Degree of biodegradation (Dt):%	
	1#	2#	3#	average	1#	2#	3#	average	1#	2#	3#	average	Cellulose	sample	Cellulose	sample
126	71.83	60.84	65.36	66.01	225.18	213.09	225.45	221.24	263.70	260.92	273.20	265.94	155.23	199.93	95.12	94.37
127	71.86	60.87	65.42	66.05	225.35	213.24	225.64	221.41	263.74	260.96	273.24	265.98	155.36	199.93	95.20	94.37
128	71.89	60.90	65.45	66.08	225.61	213.50	225.90	221.67	263.77	261.00	273.29	266.02	155.59	199.94	95.34	94.37
129	71.92	60.92	65.49	66.11	225.89	213.76	226.23	221.96	263.81	261.04	273.36	266.07	155.85	199.96	95.50	94.38
130	71.95	60.94	65.53	66.14	226.04	213.89	226.37	222.10	263.86	261.10	273.43	266.13	155.96	199.99	95.56	94.40
131	71.99	60.97	65.58	66.18	226.28	214.11	226.63	222.34	263.90	261.16	273.48	266.18	156.16	200.00	95.69	94.40
132	72.02	60.99	65.62	66.21	226.43	214.28	226.82	222.51	263.95	261.21	273.56	266.24	156.30	200.03	95.77	94.42
133	72.05	61.01	65.66	66.24	226.60	214.43	226.98	222.67	264.03	261.27	273.63	266.31	156.43	200.07	95.85	94.44
134	72.09	61.04	65.71	66.28	226.86	214.69	227.33	222.96	264.07	261.32	273.69	266.36	156.68	200.08	96.00	94.44
135	72.11	61.07	65.75	66.31	226.99	214.82	227.46	223.09	264.15	261.41	273.79	266.45	156.78	200.14	96.07	94.47
136	72.14	61.09	65.79	66.34	227.17	214.99	227.65	223.27	264.18	261.44	273.85	266.49	156.93	200.15	96.16	94.47
137	72.17	61.12	65.82	66.37	227.30	215.12	227.81	223.41	264.21	261.47	273.91	266.53	157.04	200.16	96.23	94.48
138	72.23	61.17	65.86	66.42	227.45	215.29	228.00	223.58	264.24	261.51	273.96	266.57	157.16	200.15	96.30	94.47
139	72.26	61.20	65.89	66.45	227.57	215.39	228.11	223.69	264.28	261.55	274.00	266.61	157.24	200.16	96.35	94.48
140	72.29	61.22	65.93	66.48	227.68	215.49	228.26	223.81	264.30	261.58	274.04	266.64	157.33	200.16	96.40	94.48
141	72.33	61.25	65.98	66.52	227.75	215.56	228.36	223.89	264.35	261.64	274.11	266.70	157.37	200.18	96.43	94.49
142	72.35	61.27	66.03	66.55	227.81	215.62	228.42	223.95	264.40	261.69	274.16	266.75	157.40	200.20	96.45	94.50
143	72.38	61.29	66.07	66.58	227.92	215.74	228.55	224.07	264.46	261.75	274.25	266.82	157.49	200.24	96.50	94.52
144	72.41	61.31	66.11	66.61	227.99	215.80	228.63	224.14	264.55	261.81	274.34	266.90	157.53	200.29	96.53	94.54
145	72.45	61.34	66.16	66.65	228.03	215.86	228.71	224.20	264.59	261.85	274.38	266.94	157.55	200.29	96.54	94.54
146	72.48	61.36	66.20	66.68	228.09	215.92	228.80	224.27	264.63	261.89	274.45	266.99	157.59	200.31	96.56	94.55
147	72.51	61.38	66.24	66.71	228.15	215.98	228.86	224.33	264.68	261.94	274.50	267.04	157.62	200.33	96.58	94.56
148	72.55	61.42	66.28	66.75	228.20	216.02	228.92	224.38	264.73	261.98	274.56	267.09	157.63	200.34	96.59	94.56
149	72.58	61.44	66.32	66.78	228.26	216.07	228.99	224.44	264.78	262.04	274.60	267.14	157.66	200.36	96.61	94.57
150	72.61	61.46	66.36	66.81	228.29	216.10	229.05	224.48	264.83	262.11	274.69	267.21	157.67	200.40	96.61	94.59
151	72.64	61.48	66.40	66.84	228.39	216.20	229.15	224.58	264.86	262.14	274.75	267.25	157.74	200.41	96.65	94.60
152	72.67	61.51	66.46	66.88	228.45	216.25	229.22	224.64	264.89	262.16	274.79	267.28	157.76	200.40	96.67	94.59
153	72.69	61.53	66.51	66.91	228.49	216.30	229.28	224.69	264.91	262.17	274.82	267.30	157.78	200.39	96.68	94.59
154	72.72	61.55	66.55	66.94	228.52	216.34	229.33	224.73	264.94	262.21	274.87	267.34	157.79	200.40	96.69	94.59
155	72.77	61.60	66.60	66.99	228.56	216.37	229.38	224.77	264.98	262.26	274.93	267.39	157.78	200.40	96.68	94.59
156	72.80	61.62	66.64	67.02	228.59	216.40	229.41	224.80	265.05	262.31	274.96	267.44	157.78	200.42	96.68	94.60
157	72.82	61.64	66.69	67.05	228.63	216.43	229.46	224.84	265.09	262.34	275.01	267.48	157.79	200.43	96.69	94.60
158	72.85	61.66	66.73	67.08	228.66	216.45	229.50	224.87	265.15	262.40	275.07	267.54	157.79	200.46	96.69	94.62
159	72.86	61.67	66.77	67.10	228.69	216.48	229.56	224.91	265.17	262.43	275.11	267.57	157.81	200.47	96.70	94.62
160	72.88	61.69	66.82	67.13	228.72	216.50	229.60	224.94	265.23	262.50	275.19	267.64	157.81	200.51	96.70	94.64
161	72.91	61.71	66.86	67.16	228.76	216.53	229.65	224.98	265.26	262.53	275.22	267.67	157.82	200.51	96.70	94.64
162	72.95	61.74	66.91	67.20	228.80	216.57	229.69	225.02	265.32	262.58	275.29	267.73	157.82	200.53	96.70	94.65
163	72.97	61.76	66.96	67.23	228.83	216.59	229.73	225.05	265.35	262.61	275.35	267.77	157.82	200.54	96.70	94.66
164	72.99	61.77	66.99	67.25	228.85	216.60	229.76	225.07	265.38	262.63	275.39	267.80	157.82	200.55	96.70	94.66
165	73.02	61.79	67.03	67.28	228.87	216.62	229.81	225.10	265.40	262.66	275.43	267.83	157.82	200.55	96.70	94.66

Day	Blank container				Reference material container (cellulose)				Sample container (2019E0622)				Average evolved CO_2 subtracting that of blank, g		Degree of biodegradation (Dt),%	
	1#	2#	3#	average	1#	2#	3#	average	1#	2#	3#	average	Cellulose	sample	Cellulose	sample
166	73.04	61.80	67.06	67.30	228.90	216.64	229.85	225.13	265.43	262.70	275.48	267.87	157.83	200.57	96.71	94.67
167	73.05	61.81	67.10	67.32	228.92	216.66	229.87	225.15	265.47	262.75	275.54	267.92	157.83	200.60	96.71	94.69
168	73.08	61.83	67.14	67.35	228.95	216.69	229.90	225.18	265.49	262.78	275.58	267.95	157.83	200.60	96.71	94.69
169	73.10	61.85	67.19	67.38	228.99	216.73	229.94	225.22	265.56	262.84	275.66	268.02	157.84	200.64	96.72	94.70
170	73.12	61.87	67.24	67.41	229.01	216.76	229.98	225.25	265.60	262.88	275.73	268.07	157.84	200.66	96.72	94.71
171	73.15	61.90	67.30	67.45	229.04	216.80	230.03	225.29	265.65	262.94	275.80	268.13	157.84	200.68	96.72	94.72
172	73.18	61.93	67.36	67.49	229.06	216.81	230.06	225.31	265.70	262.98	275.86	268.18	157.82	200.69	96.70	94.73
173	73.20	61.94	67.39	67.51	229.10	216.84	230.11	225.35	265.74	263.02	275.90	268.22	157.84	200.71	96.72	94.74
174	73.23	61.96	67.43	67.54	229.13	216.86	230.15	225.38	265.80	263.08	275.99	268.29	157.84	200.75	96.72	94.76
175	73.26	61.98	67.47	67.57	229.16	216.89	230.18	225.41	265.83	263.12	276.04	268.33	157.84	200.76	96.72	94.76
176	73.28	61.99	67.50	67.59	229.18	216.90	230.21	225.43	265.87	263.16	276.11	268.38	157.84	200.79	96.72	94.77
177	73.31	62.01	67.54	67.62	229.21	216.94	230.26	225.47	265.92	263.20	276.17	268.43	157.85	200.81	96.72	94.78
178	73.34	62.04	67.57	67.65	229.22	216.95	230.30	225.49	265.96	263.24	276.21	268.47	157.84	200.82	96.72	94.79
179	73.36	62.05	67.60	67.67	229.24	216.97	230.32	225.51	265.99	263.28	276.26	268.51	157.84	200.84	96.72	94.80
180	73.38	62.06	67.63	67.69	229.27	217.01	230.37	225.55	266.04	263.31	276.30	268.55	157.86	200.86	96.73	94.81

Note: It can be seen from table 5-5, at the end of testing the largest differences between the individual results of blank, reference materials and sample testing was 15.4%, 5.8%, 4.7% respectively which were all less than 20%, so calculated the average percentage biodegradation as the degree of biodegradation of material.

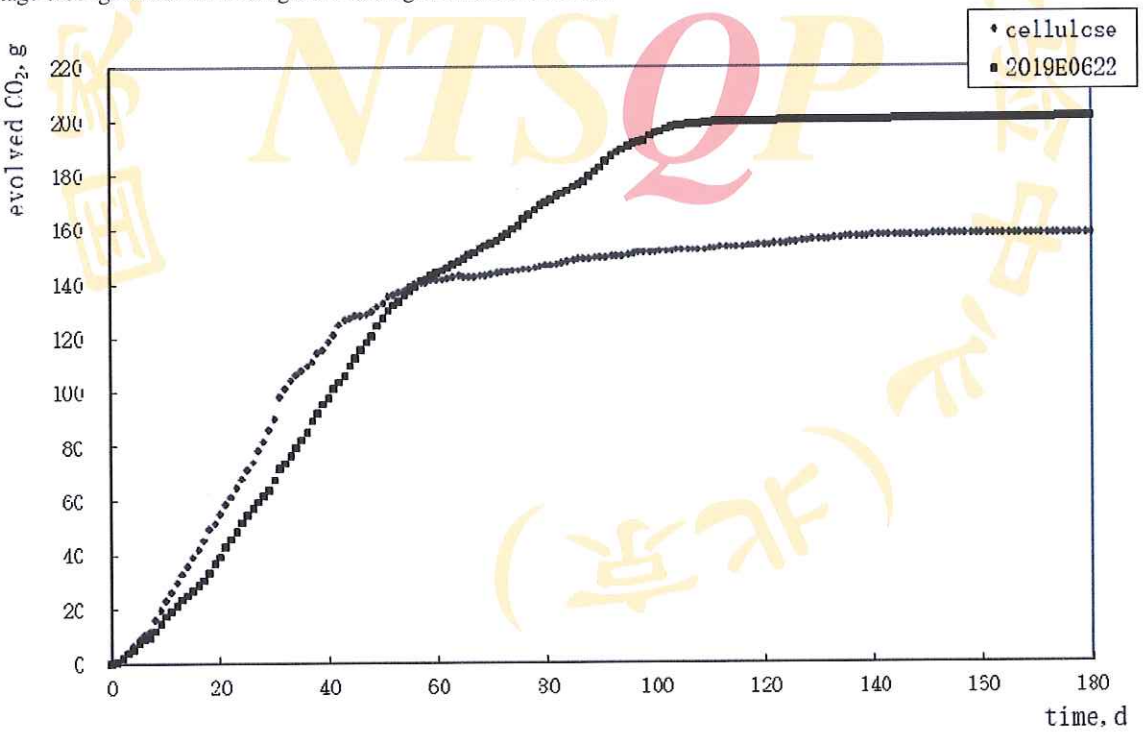

Fig. 5-2 curve of evolved CO_2

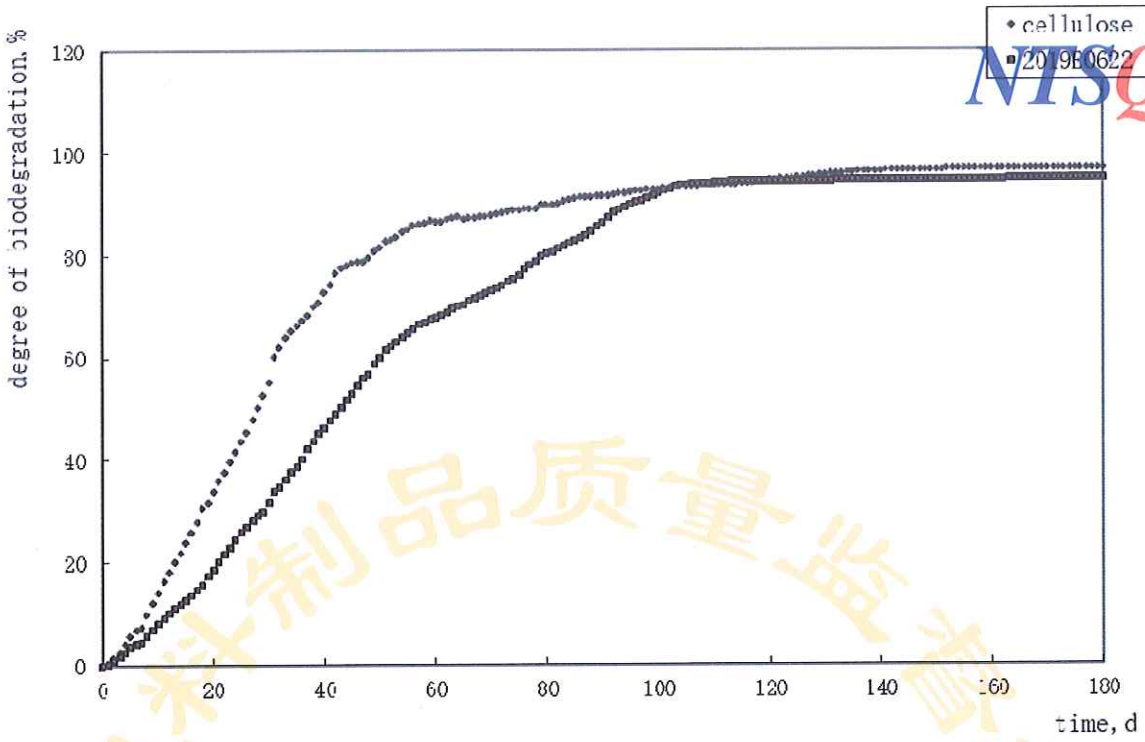

Fig. 5-3 curve of degree of biodegradation

Table 5-5, fig.5-2 and fig.5-3 showed that at the end of testing, the degree of biodegradation of cellulose as reference material and test sample (2019E0622) was 96.73% and 94.81%, respectively. Therefore, the relative degree of biodegradation of test sample corresponding to that of reference material was 98.02% rounding off the result to 98.0%.

In EN 13432, the biodegradation should be not less than 90%, the biodegradation of test sample was coincident with the requirement.

6) Analysis of valid of testing

a. Table 5-5 showed that after 10 days, the blank testing evolved about 16.12g CO_2, the content of volatile solids of 600g dry inoculum was 154.81g. So after 10 days, the inoculums in blank vessel produced 104.1mg carbon dioxide per gram of volatile solids. It was in according with the requirement in ISO 14855-1 that the inoculum in the blank shall produce between 50mg and 150mg of carbon dioxide per gram of volatile solids over the first 10 days of the test.

b. It also can be seen from table 5-5, the biodegradation of reference material was 78.6% after 45 days which required in ISO 14855-1 should be more than 70%.

c. The difference between the percentage biodegradation of the reference material in the different vessel was 5.8% at the end of the test which was less than 20% as required in standard.

So a conclusion could be made that the testing was valid and the degree of biodegradation of sample was 98.0% after 180 days.

Section 6 Disintegration testing in pilot-scale composting

6.1 Introduction

1) Principle of test method

The disintegration testing was performed under defined and standardized composting conditions on a pilot-scale level in a 170L bin. The test material was mixed with fresh biowaste, put into an insulated composting bin, then composting starts spontaneously. Monitored temperature, pH-value, moisture content and gas composition regularly. Directed the processing of composting through aeration and moisture content. The composting process was continued till fully stabilized compost obtained after 12 weeks.

2) Guidelines

ISO 16929:2002 Plastics -- Determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test

GB/T 6003.2-1997 Test sieves -- Technical requirements and testing -- Part 2: Test sieves of perforated metal plate (eqv ISO 3310-2:1990)

6.2 Materials

1) Test sample

Name: eSUN Biodegradable Polycaprolactone

Description: white sheet

2) Artificial biowaste

cabbage

fresh apple

fresh apple peel

wood chips

rabbit feed

mature compost, from the composting factory in Beijing, 3 months age. (The grade after testing with Rottegrad method was Ⅴ)

6.3 Disintegration testing

1) Apparatus

Element analyzer (precision，0.1%)

Muffle furnace (precision，±10℃)

Temperature detector (precision，±0.2%)

Moisture meter (precision，±3.0%)

Oxygen detector (precision，±3.0%)

CO_2 detector (precision，±2.5%)

pH meter (precision，±0.05pH)

Balance (precision，±0.1mg，measuring range 160g; precision，±0.1g，measuring range 610g; precision，±5g，measuring range 30kg)

Drying oven (precision，±2℃)

Disintegration bin (170L)

Sieve (2mm and 10mm)

Dewar vessels (1.3L)

Digital Camera

Reflectometer (resolution 0.1%)

- ammonium nitrogen (detection limit 0.005g/L)
- nitrite and nitrate nitrogen (detection limit 0.005g/L)

Conductivity meter(0.00~19.99mS/cm，resolution 0.01mS/cm)

2) Schematic

The schematic of apparatus was described as following fig.6-1.

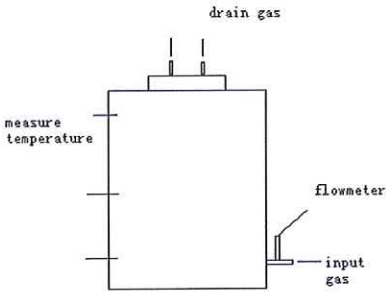

Fig.6-1 Pilot scale composting apparatus schematic

3) Testing Methods

a. Determination of the total dry solids content (TDS) and volatile solids content (VS)

Dried a crucible for 3h at 105℃, then took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_0);

Added about 3~10g sample (less than 2/3 of crucible volume) in crucible, weighed it (record as m_1);

Then put it in the drying oven and dried it for 3h at 105℃, took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_2);

Then moved it into the muffle furnace and incinerated it at 550℃ for 3h, took it out and put in the desiccator to cool to room temperature, weighed it (recorded as m_3);

Calculated the total dry solids content of sample as following:

$$TDS\ (\%) = \frac{m_2 - m_0}{m_1 - m_0} \times 100$$

calculated the volatile solids content of sample as following,

$$\mathrm{VS}\ (\%) = \frac{m_2 - m_3}{m_2 - m_0} \times 100$$

Repeated the test one time.

Determination result shall be expressed by the average value.

b. Total Organic-carbon and nitrogen content

The elements of organic carbon (TOC) and nitrogen were measured by elemental analyzer. Took about 2~5mg sample, put it into the sample cell after packed with tin boat. Started the procedure to determine elements while the temperature of combustion tube was 1150℃.

c. pH value

Determined the pH value of compost using following steps. Prepared a mixture of 1 part of compost with 5 parts of deionized water which means that 20g±0.1g compost was put into a 200ml beaker, added 100g±0.1g of deionized water, shook it, measured pH immediately with pH meter.

d. Concentration of carbon dioxide and oxygen in exhaust air.

Used oxygen and carbon dioxide detector to measure the oxygen and carbon dioxide concentration in the exhaust air respectively.

e. Moisture content

Put the detector of the moisture meter into compost and recorded the result showed on the screw of the meter.

f. Conductivity

Demarcated the detector with 2764μS/cm(2.76mS/cm) standard solution. Used conductivity meter with this detector to test the conductivity of the compost.

g. Ammonium nitrogen and nitric nitrogen

Prepared a mixture of 1 part of compost with 9 parts of deionized water which means that 4g±0.1g compost was put into a 50ml beaker, added 36g±0.1g of deionized water, shook it. Then poured solution into the reactor of Reflectometer to 5mL. Put test strip into reactor and started the test procedure, took out strip in 4min late and put into strip adapter, read the result.

h. Temperature

Tested the temperature with temperature detector and the data were transmitted to the computer.

4) Procedure

a. Sample preparation

Weighed sheet sample of 1% of biowaste (wet weight). Reduce it in size to 10cm×10cm.

Weighed granules of sample of 9% of biowaste (wet weight) to add in compost mixture to determine the effects on the composting process and the compost quality.

b. Preparation of biowaste

The following ratio of materials (wet weight) was used to prepare mixture of biowaste mixture:

—cabbage 10kg;
—fresh apple 10kg;
—fresh apple peel 0.5kg;
—rabbit feed 26kg;
—wood chips 8kg;
—mature compost 28kg
—water 30kg

Broke the above biowaste mixture to sizes not greater than 5cm and then fully mixed it. Determined the total dry solids content and volatile solids of the above respectively.

c. Prepared two bins used for blank compost testing (without test sample).

d. Prepared two bins used for sample compost testing (with test sample).

e. Feeding

Loaded test sample with biowaste into disintegration bin, and mixed it well (the moisture content about 50% by mass with no free water present).

f. Aeration

Connected air flow about 2L/min±0.03L/min at the bottom of the bin.

g. Turning

Did the turning once a week during the first four weeks, later twice a week until the end of the test to break down lumps and to remix water, micro-organisms and substrate. During turning, observed and recorded the color, odor and other characteristics of mixture and take photos for sample. Carried out a visual assessment and estimated the particle size distribution of the test material and recorded signs of microbial colonization in the test material particles.

h. Measured the pH, moisture of the mixture.

i. Oxygen and carbon dioxide concentrate

Used oxygen and carbon dioxide sensor to measure the oxygen and carbon dioxide concentration in the exhaust air respectively. Test it every working day during the first month of the test and once a week afterwards.

j. Measurement of temperature of mixture

Every morning and afternoon, measured and recorded the temperature of the biowaste mixture.

k. The duration of the incubation shall be 12 weeks.

l. Termination of the test

a) Cool the mixture to room temperature, removed it from the compost bin.

b) Sieve each of the mixture through a standard 10mm sieve, searching the overflow carefully for large lumps of the test material and breaking these up. Separate the sieved material further by sieving through a standard 2mm sieve. From the 2mm to 10mm fraction thus obtained, pick out all fragments of the test material, place them on a separate 2mm sieve and clean carefully by washing under a running tap. Dry the cleaned particles at 105℃ (or at 40℃±2℃ for test materials with melting temperatures below 105℃) until constant mass is reached. From the mass of total dry solids thus obtained, calculate the degree of disintegration as following:

$$D_i(\%) = \frac{M_{db} - M_{dt}}{M_{db}} \times 100$$

Where,

M_{db}: total dry solid mass before the test;

M_{dt}: total dry solid mass after test

In addition, measure the amount of organic matter present by determining the volatile-matter content.

c) Analyse a homogeneous sample of the ＜10mm fraction for the total dry solids, volatile solids, pH, ammonium nitrogen, nitrite and nitrate nitrogen, and total nitrogen of the mixture (＜10mm fraction) to see the quality of compost.

Determination of the maturity of the compost by the method "Rottegrad". The measured maximum temperature after about 2 to 5 days is used to classify the compost.

d) Use the ＜10mm fraction for further ecotoxicity testing.

6.4 Result and discussion

1) Basic properties of materials

a. Basic properties of biowaste

The test result of dry solids, volatile solids and C/N was listed in table 6-1.

Table 6-1 Basic properties of fresh biowaste

Item		Fruit, vegetable and rabbit feedstuff,	Wood scrap
Dry solids	%	56.6	80.2
Moisture	%	43.4	19.8
Volatile solids	%	85.7	83.1
NH_4^+-N	g/L	0.37	——
C/N		37.3	45.5

b. Basic properties of inoculum (mature compost)

The test result of dry solids, volatile solids and C/N was listed in table 6-2.

Table 6-2 Basic properties of compost

Compost (kg)	Dry solids (kg)	Moisture (kg)	Volatile solids (%)	C/N
28.0	20.6	7.4	25.2	15.5

c. Basic properties of sample

The test result of dry solids sample was listed in table 6-3.

Table 6-3 Basic properties of test sample

	Appearance ----	Dry solids (%)	Moisture (%)
Sample	Sheet	99.4	0.6

2) Test set

Table 6-4 Basic properties of mixture for composting

Composting recipient	Biowaste mixture		Test material(sample)				Ratio of carbon and nitrogen of mixture (C/N)
			Sheet sample (about 1% of biowaste)		Granule sample (about 9% of biowaste)		
	Actually added amount (kg)	Dry solids (kg)	Actually added amount (kg)	Dry solids (kg)	Actually added amount (kg)	Dry solids (kg)	
Test material (sample) recipient 1#	112.5	53.4	1.125	1.119	10.125	10.069	34.8
Test material (sample) recipient 2#	112.5	53.4	1.125	1.119	10.125	10.069	34.8
Blank recipient 1#	112.5	53.4	——	——	——	——	29.8
Blank recipient 2#	112.5	53.4	——	——	——	——	29.8

3) Temperature

The temperature of biowaste mixture in test recipients was listed in table 6-5.

Table 6-5 Temperature of test

Time, day		Blank composting recipient 1#,℃	Blank composting recipient 2#,℃	Sample composting recipient 1#, ℃	Sample composting recipient 2#, ℃
1st	am	22.4	23.6	23.5	23.6
	pm	41.9	42.1	41.5	41.7
2nd	am	52.9	51.8	53.1	52.3
	pm	56.9	62.6	62.5	58.4
3rd	am	60.7	65.1	66.8	62.3
	pm	63.5	65.9	67.7	64.2
4th	am	65.2	64.8	68.1	67.5
	pm	65.7	64.2	67.2	65.6
5th	am	64.6	66.1	66.7	66.7
	pm	65.7	64.9	65.1	67.3
6th	am	64.9	63.2	66.2	68.3
	pm	65.1	62.5	65.9	67.9
7th	am	64.7	61.9	64.2	66.5
	pm	63.8	61.1	65.1	64.8
8th	am	62.6	60.6	64.5	62.7
	pm	61.9	60.9	63.7	62.8
9th	am	62.5	61.1	63.9	60.9
	pm	61.8	61.5	63.2	60.5
10th	am	61.6	60.3	64.1	61.6
	pm	61.1	59.9	63.8	61.6
11th	am	61.0	60.8	62.6	60.8
	pm	59.7	60.7	62.1	60.8
12th	am	58.7	60.4	61.4	59.2
	pm	58.1	59.4	61.0	59.3
13th	am	57.8	59.4	59.7	57.8
	pm	57.6	59.0	59.3	58.4
14th	am	57.5	57.9	58.2	58.0
	pm	55.7	58.6	57.3	56.5
15th	am	54.7	55.8	55.3	56.1
	pm	56.4	55.5	56.2	56.8
16th	am	54.1	53.3	56.9	54.6
	pm	55.0	53.4	55.5	54.0
17th	am	53.7	52.7	55.1	53.8
	pm	55.3	52.9	54.1	52.8
18th	am	54.0	51.4	53.4	52.3
	pm	54.0	51.0	53.6	51.6
19th	am	52.7	50.2	52.8	52.6
	pm	52.2	50.2	52.1	50.7
20th	am	51.2	49.5	52.0	52.2
	pm	51.1	49.7	51.8	51.8
21st	am	50.2	48.2	51.3	51.8
	pm	50.9	48.4	50.8	51.1
22nd	am	50.0	47.6	49.0	49.5
	pm	49.2	47.4	49.5	48.5
23rd	am	49.2	47.7	50.1	48.7
	pm	48.6	46.3	48.9	48.8
24th	am	47.6	45.5	49.4	48.0
	pm	47.2	44.7	50.9	49.5
25th	am	47.0	44.3	47.2	47.4
	pm	47.1	44.9	46.7	48.2
26th	am	46.3	43.5	45.7	45.4
	pm	45.9	42.9	45.7	46.9
27th	am	44.5	42.8	43.0	41.5
	pm	47.2	41.6	43.3	41.9
28th	am	43.7	40.9	40.5	41.6
	pm	41.9	41.0	41.4	40.8

Time, day		Blank composting recipient 1#,℃	Blank composting recipient 2#,℃	Sample composting recipient 1#, ℃	Sample composting recipient 2#, ℃
29th	am	42.8	41.1	42.3	41.6
	pm	43.0	39.6	41.6	40.7
30th	am	41.1	39.1	40.9	41.2
	pm	41.2	39.6	40.0	40.9
31st	am	39.3	39.3	40.5	39.4
	pm	39.1	39.3	40.3	39.0
32nd	am	39.0	39.2	40.2	38.7
	pm	39.4	39.3	39.1	39.3
33rd	am	39.5	39.5	39.6	38.8
	pm	38.5	38.7	38.4	38.6
34th	am	39.1	38.2	37.9	38.5
	pm	37.3	38.1	36.4	37.2
35th	am	35.7	36.7	35.8	36.0
	pm	38.7	37.9	38.3	37.1
36th	am	36.1	36.9	35.9	37.8
	pm	36.5	36.2	36.8	38.2
37th	am	35.2	36.0	35.8	34.7
	pm	37.6	37.1	36.9	36.1
38th	am	35.3	35.0	35.7	36.0
	pm	35.1	35.8	36.2	33.6
39th	am	35.3	35.0	34.1	34.1
	pm	34.7	34.5	34.4	35.6
40th	am	34.5	34.4	36.8	36.0
	pm	34.7	33.2	33.1	34.7
41st	am	33.4	33.5	33.2	35.2
	pm	34.5	34.5	33.9	34.6
42nd	am	33.0	32.2	33.7	33.5
	pm	32.9	33.4	32.0	32.9
43rd	am	32.8	32.0	32.4	32.4
	pm	31.7	31.4	31.5	30.7
44th	am	31.8	32.6	31.8	30.7
	pm	33.0	32.4	30.8	32.6
45th	am	31.3	32.0	31.2	31.9
	pm	30.7	31.5	31.1	31.7
46th	am	31.5	31.9	31.8	32.6
	pm	30.5	29.2	28.9	30.3
47th	am	31.0	30.7	32.7	30.9
	pm	29.8	29.8	29.6	29.0
48th	am	29.8	28.7	30.3	29.4
	pm	30.9	30.6	31.9	31.0
49th	am	29.7	29.8	29.4	30.6
	pm	29.6	28.6	30.0	31.1
50th	am	30.7	29.6	30.8	29.6
	pm	31.7	31.4	30.9	31.1
51st	am	28.8	29.0	27.6	28.4
	pm	28.9	28.6	27.8	29.4
52nd	am	27.4	27.8	27.8	26.7
	pm	27.8	26.3	25.6	25.6
53rd	am	27.5	26.2	26.7	25.7
	pm	26.3	26.1	24.9	25.5
54th	am	24.6	24.7	25.8	23.4
	pm	24.7	25.4	24.9	24.6
55th	am	24.5	23.4	25.5	24.8
	pm	24.5	24.7	24.9	24.2
56th	am	25.4	26.1	24.1	24.1
	pm	25.7	25.7	26.6	24.8
57th	am	25.3	24.1	24.7	26.6
	pm	25.0	24.7	26.0	25.3
58th	am	24.4	24.2	26.3	26.8
	pm	24.9	24.6	24.6	25.5
59th	am	24.7	25.4	25.3	26.1

Time, day		Blank composting recipient 1#,℃	Blank composting recipient 2#,℃	Sample composting recipient 1#, ℃	Sample composting recipient 2#, ℃
	pm	25.0	24.0	24.7	24.8
60th	am	24.6	25.6	25.6	24.3
	pm	25.0	23.8	23.8	25.5
61st	am	24.8	24.7	25.7	26.9
	pm	24.8	25.3	26.3	25.7
62nd	am	25.9	24.5	23.3	24.3
	pm	24.0	24.3	22.7	24.3
63rd	am	24.6	24.5	25.2	25.4
	pm	23.9	23.8	24.6	24.1
64th	am	24.7	24.4	25.0	24.7
	pm	24.2	24.1	25.8	25.1
65th	am	24.3	24.7	24.3	22.5
	pm	24.7	24.1	25.2	25.8
66th	am	23.8	24.5	25.0	22.6
	pm	25.7	24.9	24.7	24.0
67th	am	23.8	24.3	25.6	25.2
	pm	24.2	24.8	24.0	25.6
68th	am	24.6	23.6	24.7	24.9
	pm	24.1	23.6	25.9	24.5
69th	am	24.9	24.1	25.3	23.4
	pm	25.0	23.7	23.4	25.0
70th	am	24.1	24.3	25.2	24.3
	pm	23.9	23.7	24.1	24.5
71st	am	23.7	23.4	25.8	24.7
	pm	24.7	23.4	24.4	25.1
72nd	am	23.7	22.9	24.5	23.7
	pm	24.6	23.6	24.5	24.6
73rd	am	24.9	24.2	23.5	23.6
	pm	24.6	23.9	22.8	24.5
74th	am	23.3	24.2	24.2	23.5
	pm	23.9	24.6	23.2	24.7
75th	am	24.0	23.6	23.8	23.1
	pm	23.6	23.8	24.1	23.8
76th	am	24.1	23.8	23.9	23.8
	pm	24.0	23.8	24.2	24.1
77th	am	23.8	23.1	23.2	23.3
	pm	24.4	23.4	24.8	24.5
78th	am	23.5	23.2	23.2	23.8
	pm	23.9	23.7	23.4	24.1
79th	am	24.5	23.6	23.3	24.3
	pm	24.4	24.1	22.9	24.3
80th	am	24.1	24.4	24.3	24.2
	pm	23.5	23.3	23.7	23.8
81st	am	23.7	23.4	23.8	23.8
	pm	23.2	23.5	23.5	23.6
82nd	am	23.5	24.5	23.8	24.1
	pm	23.6	23.7	24.0	23.7
83rd	am	23.8	23.8	23.5	23.5
	pm	23.9	23.7	23.2	23.8
84th	am	23.4	23.5	23.2	24.1
	pm	23.9	23.8	23.8	23.9

It can be seen from table 6-5, in the first week, the highest temperature of the test recipients was 65.7℃, 66.1℃, 68.1℃ and 68.3℃ respectively which lower than 75℃ and it decreased after one week thereafter.

The temperature of all recipients remained above 60℃ about one week. The temperature of blank recipient 1# remained above 40℃ for 29.5 consecutive days while blank recipient 2# remained for 28.0 days. And the temperature of test sample recipient 1# remained above 40℃ for 31.0 consecutive days while test recipient 2# remained for 29.5 days. It can be seen that all recipients had the times above 40℃ more than 4 consecutive weeks. It seems all right of test characters of composting, so it considered the processing of test

were valid.

4) Oxygen and Carbon dioxide concentrate

Fig.6-2 and fig.6-3 was the curve of carbon dioxide and oxygen concentration related with the composting time respectively.

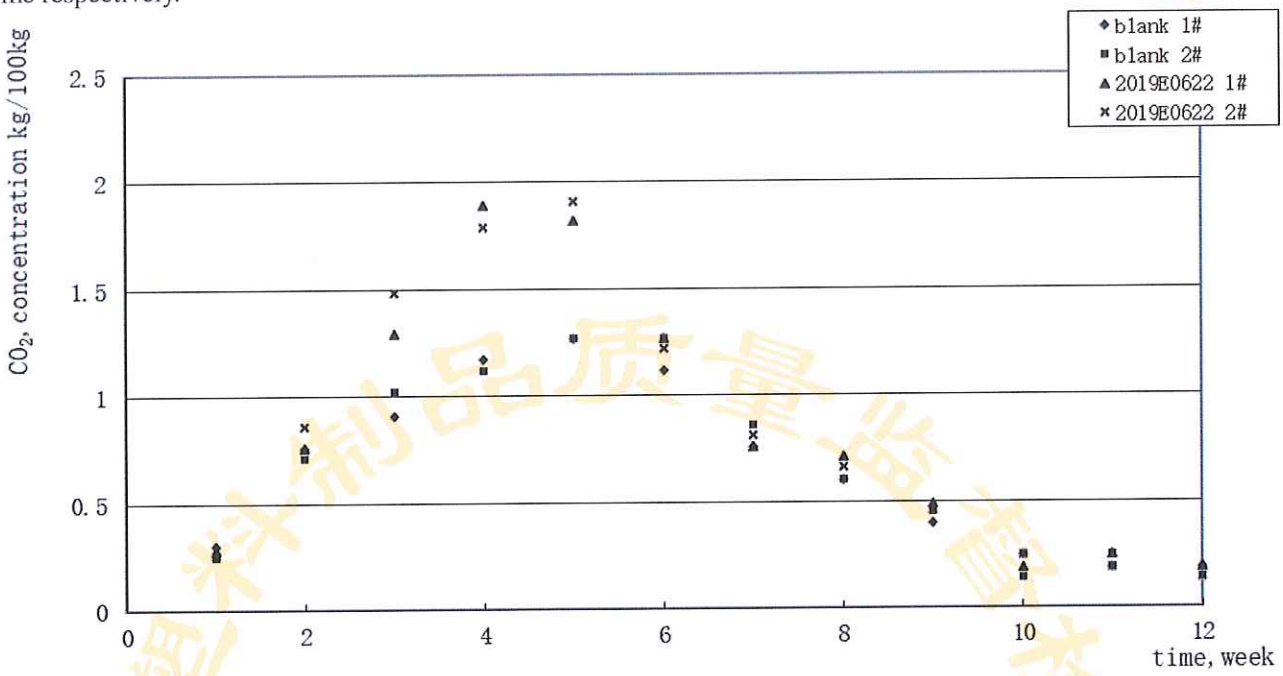

Fig. 6-2 CO_2 concentration curve

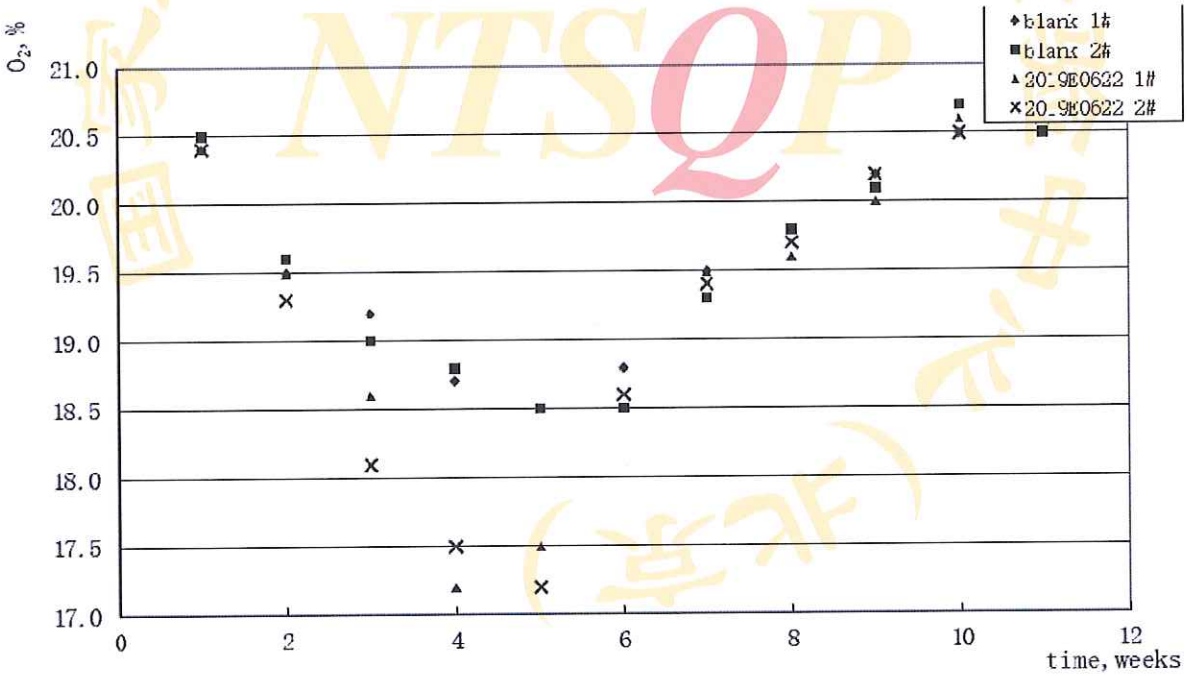

Fig.6-3 oxygen concentration curve

From fig.6-2, it can be seen that the percentage of concentration of carbon dioxide in exhaust air was increased quickly in the firstly 4 weeks with composting processing and decreased after 5 weeks and after 10 weeks it changed slowly. It also can be seen from fig.6-3 that in the exhaust air, the concentration of oxygen of each recipient was more than 17.0% which large than 10% as required in standard to ensure the composting going on well.

5) pH

pH of each test recipient was listed on table 6-6.

Table 6-6 pH value

Weeks	Blank composting recipient		Sample composting recipient	
	1#	2#	1#	2#
0	7.0	7.0	7.0	7.0
1	7.1	7.0	7.1	7.1
2	7.1	7.1	7.0	7.1
3	7.2	7.2	7.1	7.2
4	7.3	7.3	7.2	7.2
5	7.3	7.3	7.4	7.3
6	7.4	7.4	7.5	7.3
7	7.5	7.4	7.5	7.5
8	7.5	7.5	7.6	7.5
9	7.6	7.6	7.6	7.6
10	7.6	7.6	7.7	7.6
11	7.7	7.6	7.7	7.7
12	7.8	7.7	7.6	7.7

It can be seen from table 6-6, the pH value of each vessel was 7.0 at the beginning of test. The pH value of each vessel was increased with the testing going on and the highest value was 7.8 until the test was finished. All of pH value of each vessel was lower than 9.0 and above 5.0.

6) NH_4^+-N, NO_x^--N, Electric conductivity

The test result of NH_4^+, NO_x^- and Electric conductivity was listed on table 6-7, table 6-8 and table 6-9 respectively.

Table 6-7 NH_4^+-N in the mixture

weeks	NH_4^+-N, g/kg TDS			
	Blank compost1#	Blank compost2#	Sample compost1#	Sample compost2#
0	0.38	0.40	0.40	0.44
2	0.42	0.44	0.48	0.52
4	0.34	0.32	0.46	0.54
6	0.30	0.35	0.41	0.43
8	0.22	0.27	0.39	0.38
10	0.18	0.16	0.23	0.22
12	0.15	0.12	0.20	0.18

Table 6-8 NO_x^- in the mixture

weeks	NO_x^-, g/kg TDS			
	Blank compost1#	Blank compost2#	Sample compost1#	Sample compost2#
0	0.14	0.13	0.16	0.22
2	0.19	0.18	0.19	0.20
4	0.24	0.22	0.25	0.24
6	0.23	0.24	0.26	0.30
8	0.29	0.27	0.34	0.30
10	0.29	0.27	0.31	0.29
12	0.29	0.26	0.30	0.28

Table 6-9 E.C. in the mixture

weeks	E.C., ms/cm			
	Blank compost1#	Blank compost2#	Sample compost1#	Sample compost2#
0	2.21	2.24	2.20	2.21
2	2.08	2.12	2.04	1.95
4	1.99	1.96	1.83	1.87
6	1.84	1.79	1.82	1.82
8	1.63	1.76	1.70	1.75
10	1.63	1.64	1.68	1.69
12	1.58	1.61	1.65	1.67

Table 6-7 showed that the concentration of NH_4^+ of blank compost mixture and sample compost mixture was decreased with processing of composting and it was 0.15g/kg, 0.12g/kg, 0.20g/kg, 0.18g/kg at the end of

the testing respectively.

Table 6-8 showed that the concentration of NO_x^- of blank compost mixture was increased with processing of composting(1# to 0.29g/kg TDS; 2# to 0.26g/kg TDS), and the concentration of NO_x^- of sample compost mixture was also increased(1# to 0.30g/kg TDS; 2# to 0.28g/kg TDS).

Table 6-9 showed that the value of E.C. of blank compost mixture and sample compost mixture was decreased after beginning test.

It can be seen from table 6-7 to table 6-9, the processing of composting went smoothly.

7) Characteristics of the compost after composting test

Analyse a homogeneous mixture of the <10mm fraction for total dry solids, volatile solids, C/N and Rottegrad. The result was listed on table 6-10.

Table 6-10 Characteristics of the compost after composting

	Dry solids (%)		Volatile solids (%)		C/N		Rottegrad	
	1#	2#	1#	2#	1#	2#	1#	2#
Blank compost after testing	51.4	51.8	15.2	15.0	16.1	15.7	V	V
Sample compost after testing	51.9	51.9	15.8	15.5	16.9	17.1	V	V

From table 6-10, it can be seen that the compost mixture was mature at the end of testing which can be used to do the ecotoxicity evaluation.

8) Visual description

At first, put the sample into the biowaste which mixtured by the proper proportion. After turning them, begin the test.

The visual photo of residual sample in composting processing in different weeks until out of sight were listed from fig.6-4 to fig.6-7.

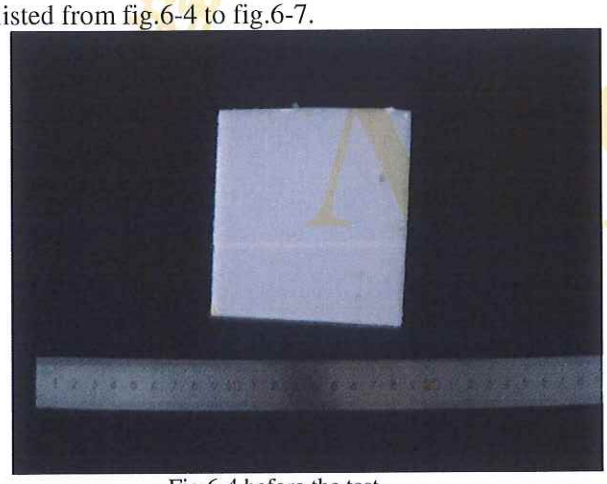

Fig.6-4 before the test

Fig.6-5 sample in the first week

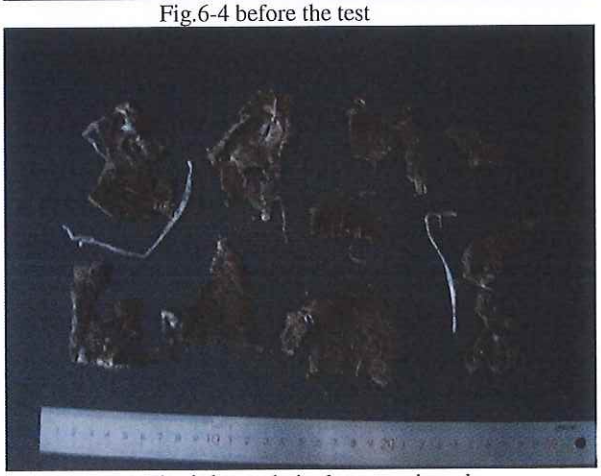

Fig.6-6 sample in the second week

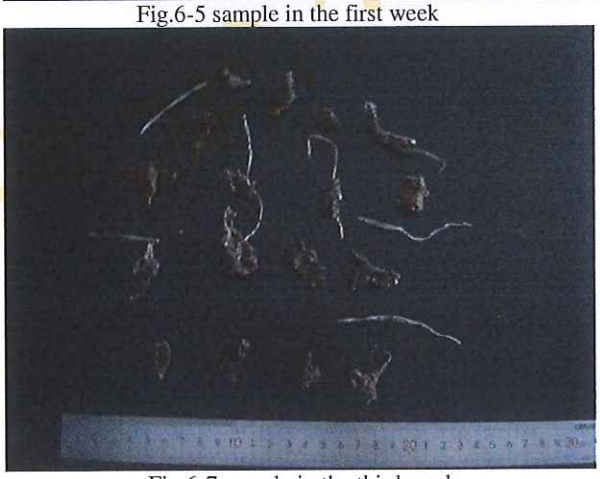

Fig.6-7 sample in the third week

From fig.6-4 to fig.6-7, it can be seen that the shape of residual sample in the composting became smaller. The reason was the sample was disintegrated and the organic carbon of the sample was degraded to dioxide carbon. In the initial two weeks the changes of sample was not obvious. After the third week, the sample was disintegrated to small fragments, it nearly not distinguished residual sample from compost. Then, there were no significant changes in the appearance of the mixture.

At the end of test, picked out the sample, and then sieved the compost mixture through a standard 10mm sieve and 2mm sieve respectively. The compost was listed on fig.6-8 and fig.6-9 after sieved. Later, inspected rudimental sample. Dried the cleaned fragments of sample at 105℃ after washing, and recoded the value which was the mass of total dry solids of residual sample.

Fig.6-8 compost through 10mm sieve

Fig.6-9 compost through 2mm sieve

9) Degree of disintergration

The residual test sample was the fragments from sample compost after testing that fail to pass through a > 2mm fraction sieve. The amount of dry solids in the residual test sample was determined and listed on table 6-11. The result of disintegration.

Table 6-11 the result of disintegration

Number of apparatus	The amount of total dry solids in the test sample input(g)	The amount of total dry solids in the residual test sample(g)	The degree of disintegration(%)
1#	1118.8	0.1	100.0
2#	1118.8	0.1	100.0
Average			100.0

From table 6-11, it can be seen that the average disintegration was 100.0%, which was corresponding the requirement of 90% in EN 13432.

Section 7 Ecotoxicity

7.1 Introduction

1) Principle of test method

The plant growth testing was done with the compost after disintergration test. The compost <10mm fraction at the end of the composting may contain residual of test sample such as metabolites, undegraded components and inorganic components. The purpose of the plant testing was to evaluate any toxic effect of the products of composting to the plant compared with blank compost.

At the end of test, the germination rate, fresh and dry weight of the plants of each pot were determined. The result of test was included the rate of germination ratio and biomass as percent of the corresponding values obtained with the blank compost.

2) Guidelines

ASTM D 6400-12 Standard Specification for Labeling of Plastics Designed to be Aerobically Composted in Municipal or Industrial Facilities

EN 13432 packaging - requirements for packaging recoverable through composting and biodegradation - Test scheme and evaluation criteria for the final acceptance of packaging

OECD 208 Guidelines for the testing of chemicals "Terrestrial plant test: seedling emergence and seedling growth test"

7.2 Materials

1) compost

a. blank compost

The blank compost was the compost of blank recipients respectively after the composting testing as described in section 6, recorded as blank compost 1# and blank compost 2#, and homogenous blank compost 1# and blank 2# as 50:50 mixture, recorded as blank compost 3#.

b. test sample compost

The test sample compost was the compost of test sample recipients respectively after the composting testing as described in section 6, recorded as sample compost 1# and sample compost 2#, and homogenous sample compost 1# and sample 2# as 50:50 mixture, recorded as sample compost 3#.

2) soil

Sampled the soil from field. Removed the surface materials such as leaves etc. on the field soil then took soil. Sieved the soil with 2mm sieve. After sieving, spread soil on the tarpaulin under sunshine with well ventilated for 3 days. Determined it's value of pH, content of organic carbon, N, conductivity and water holding capacity. The value of pH of soil should be 5.5 to 7.0.

3) seeds

2 kinds of seeds were selected from the categories of annex1 of OECD 208 listed as following.

a. Chinese cabbage of species of Brassica campestris var. chinensis in DICOTYLEDONAE

b. Barley of species of Hordeum vulgare

7.3 Planting

1) Apparatus

a. Garden pot (3.5L)

b. Balance (precision, ±0.1mg)

c. Air conditioner (precision, ±2℃)

d. Automatic humidifier (precision, ±5%RH)

e. Illumination (precision, CCT: 2000K; Initial lumens 56500)

f. Illumination detector (precision, 2.0%)

g. Temperature meter (precision, ±0.5℃)

h. Moisture meter (precision, ±3%)

i. Drying oven (precision, ±2℃)

j. Digital Camera

2) Testing method

a. Weight determination

a) fresh weight of plant

At the end of testing, weight a beaker recorded as M_{p1}(g), push the plants in a beaker and weight it recorded as M_{p2}(g). The fresh weight of plants(g)=M_{p2}-M_{p1}.

b) dry weight of plant

Dry the plants and dry it at 105℃, weigh it recorded as M_{p3}. The dry weight of plant(g)=M_{p3}-M_{p1}.

b. Germination ratio

Recorded the number of sowed seeds. At the end of test, counted the germination of plants. The germination ratio (%) = The number of germinate/sowed seeds ×100%.

c. Procedure

a) Determined the total dry solids of reference soil and compost. Each seed was planted with reference soil, compost mixed with reference soil(1:1), compost mixed with reference soil(1:3) with parallel groups respectively. Weighed reference soil or compost/reference soil (about 4000g), put it into a 3500mL pot, add about 1100mL distilled water.

b) The 2 kinds seeds had been sown as following:

Put 100 seeds of summer barley onto soil mixture in pot for each sample series.

Put 100 seeds of Chinese cabbage onto soil mixture in pot for each sample series.

Then covered the pot with a thin layer of reference soil, and then added the appropriate distilled water respectively, as needed to make the right humidity.

c) Covered the pots with glass plate, incubate them at a constant temperature of 20℃±2℃ in the dark. After germination, removed the glass, exposed the pots under at least 3000lux light shining for 16h per day. Exchanged the location of each pot every two days.

d) During the test, used automatic humidifier and air conditioner to maintain temperature 23℃±2℃ and humidity (70% during the day, at 90% in night).

e) During the test, water is added if need to the right humidity of pots.

f) Took pictures once a week, record plant growth.

g) The test was finished 14~21 days after 50% of the control plants have emerged.

h) For each species, calculated for each series of tests the germination rate. The result was expressed by the average value of parallel group.

Weigh each series test weight of each plant biomass (fresh weight). The result was expressed by the average value of parallel group.

Dry plants at 105℃, after 3 hours weigh it.

Calculate the germination ratio and biomass as percent of the corresponding values obtained with the blank compost. For each plant, calculated as follows:

*Ge*mination rate as percentage of corresponding

$$\text{values obtained with blank compost}_{(\text{compost: reference soil, 1:1})}(\%) = \frac{\text{Gemination rate}_{(\text{sample compost : reference soil, 1:1})}}{\text{Gemination rate}_{(\text{blank compost: reference soli, 1:1})}} \times 100$$

*Ge*mination rate as percentage of corresponding

$$\text{values obtained with blank compost}_{(\text{compost : reference soil, 1:3})}(\%) = \frac{\text{Gemination rate}_{(\text{sample compost : reference soil, 1:3})}}{\text{Gemination rate}_{(\text{blank compost: reference soli, 1:3})}} \times 100$$

$$\text{Biomass rate}_{(\text{compost: reference soil, 1:1})}(\%) = \frac{\text{Dry biomass}_{(\text{sample compost: reference soil, 1:1})}}{\text{Dry biomass}_{(\text{blank compost: reference soil, 1:1})}} \times 100$$

$$\text{Biobass rate}_{(\text{compost reference soil, 1:3})}(\%) = \frac{\text{Dry biomass}_{(\text{sample compos: reference soil, 1:3})}}{\text{Dry biomass}_{(\text{blank compost: reference soil, 1:3})}} \times 100$$

7.4 Result and discussion

1) The character of soil and composting-soil

the character of soil and composting-soil has been shown on following table 7-1, table 7-2 and table 7-3.

Table 7-1 Mass of the mixture in pot

Compost-soil rate	Blank series (kg)		Sample series (kg)	
	Compost	Soil	Compost	Soil
1:1	2.0	2.0	2.0	2.0
1:3	1.0	3.0	1.0	3.0

Table 7-2 Characteristics of the mixture of the [(blank-compost)+soil]

Compost-soil rate	Dry solids（%）	Moisture（%）	Volatile solids（%）	Ratio of carbon and nitrogen（C/N）
1:1	67.42	32.58	21.52	18.21
1:3	73.52	26.48	23.90	19.18

Table 7-3 Characteristics of the mixture of the [(sample-compost)+soil]

Compost-soil rate	Dry solids（%）	Moisture（%）	Volatile solids（%）	Ratio of carbon and nitrogen（C/N）
1:1	68.44	31.56	21.55	18.69
1:3	74.04	25.96	23.90	19.41

2) Test series

a. Cabbage

The series of planting of cabbage were listed on table 7-4.

Table 7-4 Test series of cabbage planting test

Test set		Numbers of seeds at the beginning of testing	Numbers of germination at the end of testing	Average
[(blank-compost)+soil]1:1	1#	100	97	97.67
	2#	100	98	
	3#	100	98	
[(blank-compost)+soil]1:3	1#	100	98	97.67
	2#	100	98	
	3#	100	97	
[(sample-compost)+soil]1:1	1#	100	97	97.33
	2#	100	97	
	3#	100	98	
[(sample-compost)+soil]1:3	1#	100	97	95.67
	2#	100	94	
	3#	100	96	

b. Barley

The series of planting of summer barley were listed on table 7-5.

Table 7-5 Test series of summer barley planting test

Test set		Numbers of seeds at the beginning of testing	Numbers of germination at the end of testing	Average
[(blank-compost)+soil]1:1	1#	100	99	98.67
	2#	100	99	
	3#	100	98	
[(blank-compost)+soil]1:3	1#	100	97	98.00
	2#	100	98	
	3#	100	99	
[(sample-compost)+soil]1:1	1#	100	97	95.67
	2#	100	96	
	3#	100	94	
[(sample-compost)+soil]1:3	1#	100	99	98.67
	2#	100	99	
	3#	100	98	

3) Rate of germination ratio

Read the average germination ration of each plant from table 7-4 to table 7-5 which was listed on table 7-6 respectively.

Table 7-6 The average germination ratio of plants

Average germination ratio		cabbage, %	barley, %
[(blank-compost)+soil]	1:1	97.7	98.7
	1:3	97.7	98.0
[(sample-compost)+soil]	1:1	97.3	95.7
	1:3	95.7	98.7

Table 7-7 was the rate of germination ratio of each plant which was calculated from the average germination ratio of plants. From table 7-7, it can be seen that the rate of germination ration of each plant was all more than 90% as required in standard.

Table 7-7 The rate of germination ratio of plants

Rate of germination ratio		cabbage, %	barley, %
[(sample-compost)+soil]/[(blank-compost)+soil]	1:1	99.6	97.0
	1:3	98.0	100.7

4) Biomass rate

a.Cabbage

The biomass of planting of cabbage were listed on table 7-8.

Table 7-8 Test biomass of cabbage planting test

Test set		Weight of fresh biomass,g	average	Weight of dry biomass,g	Average
[(blank-compost)+soil]1:1	1#	17.55	17.24	2.27	2.28
	2#	17.83		2.41	
	3#	16.35		2.16	
[(blank-compost)+soil]1:3	1#	18.23	17.51	2.19	2.18
	2#	16.99		2.10	
	3#	17.32		2.24	
[(sample-compost)+soil]1:1	1#	16.68	16.99	1.98	2.21
	2#	17.33		2.39	
	3#	16.97		2.26	
[(sample-compost)+soil]1:3	1#	17.62	18.05	2.21	2.32
	2#	18.67		2.49	
	3#	17.87		2.25	

b.Summer barley

The biomass of planting of summer barley were listed on table 7-9.

Table 7-9 Test biomass of summer barley planting test

Test set		Weight of fresh biomass,g	average	Weight of dry biomass,g	Average
[(blank-compost)+soil]1:1	1#	18.12	17.89	2.20	2.15
	2#	17.71		2.14	
	3#	17.83		2.11	
[(blank-compost)+soil]1:3	1#	17.81	17.41	2.11	2.11
	2#	17.74		2.17	
	3#	16.67		2.05	
[(sample-compost)+soil]1:1	1#	17.85	18.02	2.15	2.20
	2#	18.20		2.25	
	3#	18.02		2.21	
[(sample-compost)+soil]1:3	1#	17.16	17.28	1.99	2.02
	2#	17.13		1.96	
	3#	17.55		2.11	

Table 7-8 to table 7-9 was listed the fresh and dry weight of plants at the end of testing respectively. And the biomass rate of plants was calculated based on the dry biomass of all plants of each series which was listed on table 7-10 and table 7-11. It can be seen from table 7-12 that the biomass rate was all more than 90%.

Table 7-10 The weight of fresh plants

weight of biomass		Cabbage, g	barley, g
[(blank-compost)+soil]	1:1	17.24	17.89
	1:3	17.51	17.41
[(sample-compost)+soil]	1:1	16.99	18.02
	1:3	18.05	17.28

Table 7-11 The weight of dry biomass of plants

weight of dry biomass		Cabbage, g	barley, g
[(blank-compost)+soil]	1:1	2.28	2.15
	1:3	2.18	2.11
[(sample-compost)+soil]	1:1	2.21	2.20
	1:3	2.32	2.02

Table 7-12 The biomass rate of dry plants

Biomass rate		cabbage, %	barley, %
[(sample-compost)+soil]/[(blank-compost)+soil]	1:1	96.9	102.3
	1:3	106.4	95.7

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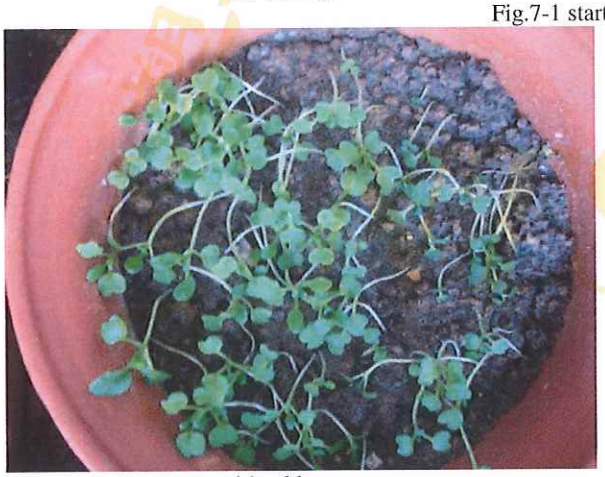
5) Visual result

The visual observations of growing of plants were taken by photos by weeks and in which one group of plants of [(sample-compost)+soil] were listed on fig.7-1 to fig.7-3.

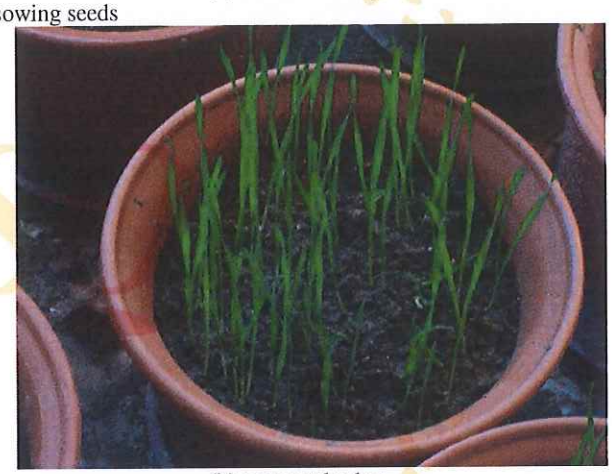
(a) cabbage

(b) summer barley

Fig.7-1 start sowing seeds

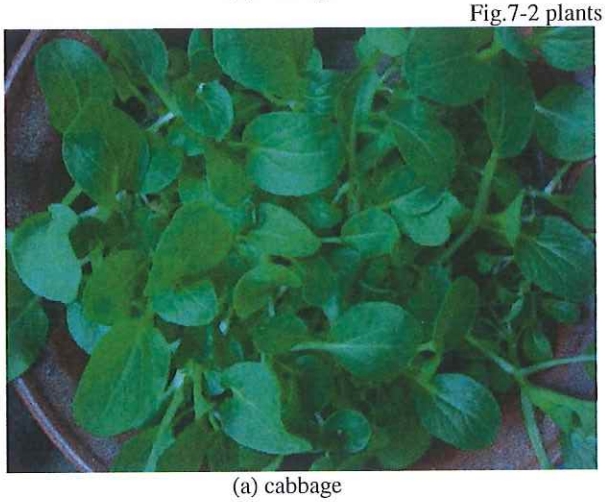


(a) cabbage

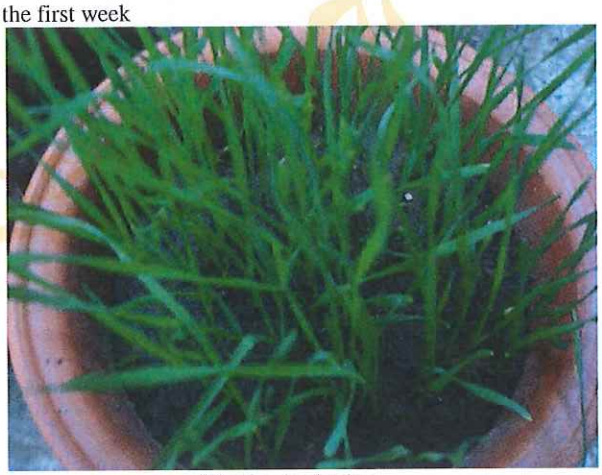


(b) summer barley

Fig.7-2 plants in the first week



(a) cabbage



(b) summer barley

Fig.7-3 plants in the third week

At beginning of testing, sowed seeds on soil mixed with compost in pot and covered evenly with a thin

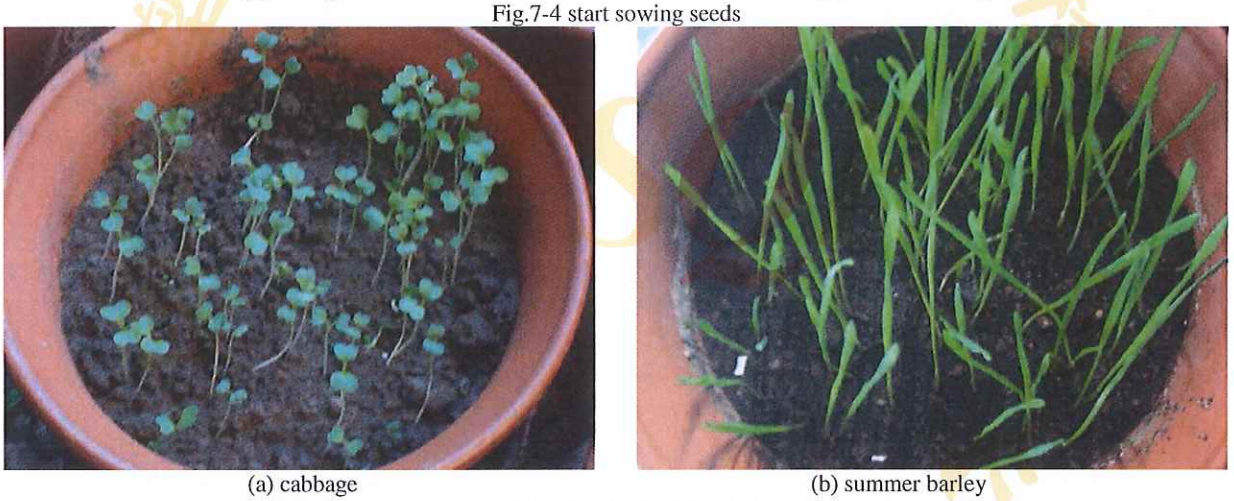
layer reference materials. Added with appropriate amount of water, thereby, the moisture of the soil maintained around in 40%. After germination, there was no one performance with yellow necrosis. In the period of planting, there was no chlorosis, putrescence, wilt, malformation of leaf and caudex in all of the test series.

The visual observations of plants during the testing showed that there were no visual difference in growth between the sample-compost mixture and blank-compost mixture.

The visual observations of growing of plants were taken by photos by weeks and in which one group of plants of [(blank-compost)+soil] were listed on fig.7-4 to fig.7-6.

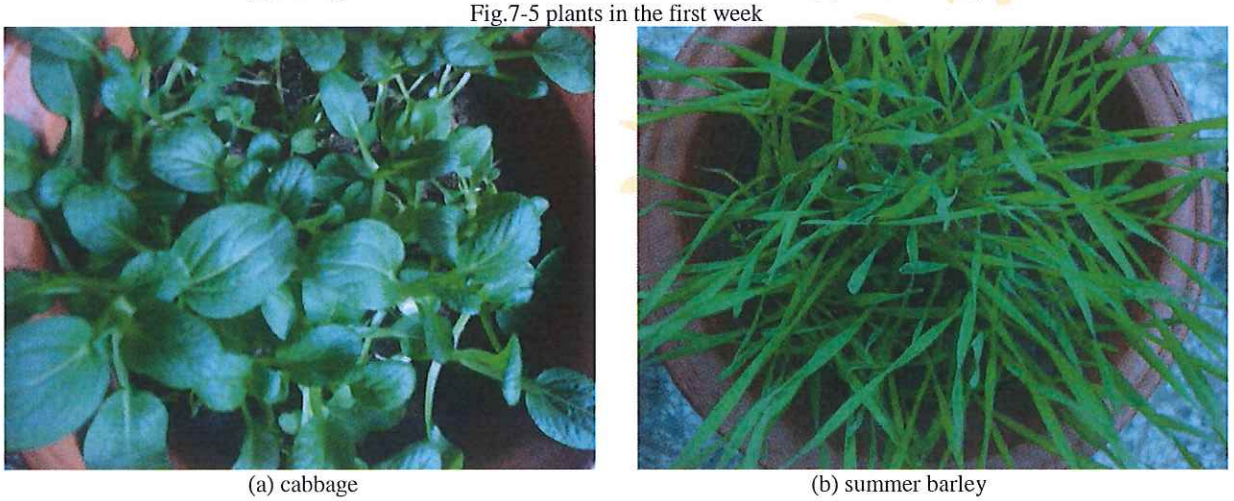
(a) cabbage (b) summer barley

Fig.7-4 start sowing seeds



(a) cabbage (b) summer barley

Fig.7-5 plants in the first week



(a) cabbage (b) summer barley

Fig.7-6 plants in the third week

6) Conclusion of the result

Table 7-7 and table 7-12 showed the rate of germination ratio and the rate of biomass of plants were all more than 90% as required in EN 13432 respectively. From above results, it can be concluded that the rate of germination ratio and biomass rate was reached the requirement of ecotoxicity of EN 13432.

————End————

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