FINAL REPORT

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

ENVIRONMENTAL DIVISION LABORATORY, MUMBAI

INTERTEK INDIA PRIVATE LIMITED

Client: ITC LIMITED PAPERBOARDS & SPECIALTY PAPERS DIVISION

Sample registration date:09/09/2022

Analysis starting date: 09/09/2022 **Analysis completed on:** 09/05/2023

Name of product: Filobev / Filotub

Quantity received and packing: - 1 packet

Sample details: Filobev / Filotub Lab reference no.15739824

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

Sampling done by: Sample not drawn by Intertek

Report No. MUM/003115B3/2022

LABORATORY

Testing as presented in this report was conducted by Environmental division of Intertek India Private Limited. The testing facility is located at F wing, 2nd Floor, Chandivali Saki vihar Road, Andheri (East), Mumbai – 400 072, India.

SAMPLE RECEIPT

The sample was received on 09/09/2022 at the Intertek testing facility. The sample was sent through courier. Sample was at ambient temperature in good condition with no evidence of damage or contamination. No temperature preservation was required.

SAMPLE DESCRIPTION:

Figure 1: Filobev / Filotub

PROCEDURE:

Filobev / Filotub samples were submitted by ITC Limited Paperboards & Specialty Papers Division for testing under EN-13432.

To determine the Compostability of materials as per EN 13432 (2000-12) is by addressing four characteristics:

1) biodegradability

2) disintegration during biological treatment,

3) effect on the biological treatment process and

4) effect on the quality of the resulting compost.

REQUIREMENTS:

1. **Characterization:**

Packaging material under testing should be identified and characterized prior to testing:

- Information and identification
- Determination of the presence of hazardous substance such as heavy metal.
- Determination of organic content, total dry solids and volatile solids of the packaging material used for biodegradation and disintegration.

2. **Biodegradation:**

Biodegradation under controlled composting condition is carried out using ISO 14855:1

3. **Disintegration during composting:** A plastic product or material will disintegrate during composting such that any remaining plastic residuals are not readily distinguishable from the other organic materials in the finished product. Additionally, the material or product must not be found insignificant quantities during screening prior to final distribution of the compost.

4. **No Adverse Impacts on Ability of Compost to Support Plant Growth**—The tested materials shall not adversely impact on the ability of composts to support plant growth, when compared to composts derived from bio waste without any addition of tested products or reference materials. Plant Growth test as per OECD 208 should be used as described in Annex E.

CRITERIA:

- **Disintegration during Composting**—A plastic product is considered to have demonstrated satisfactory disintegration if after twelve weeks (84 days) in a controlled composting test, no more than 10 % of its original dry weight remains after sieving on a 2.0-mm sieve.
- **Ultimate Aerobic Biodegradation**—A plastic product must demonstrate a satisfactory rate and level of biodegradation by achieving the following ratio of conversion to carbon dioxide given below(a) within 180 days. For all polymers, Ninety percent (90 %) of the organic carbon shall be converted to carbon dioxide by the end of the test period when compared to the positive control. Both the positive control, and test sample to be composted for the same length of time
- **Eco-toxicity—** A plastic product can demonstrate satisfactory terrestrial safety if it fulfills the requirements below

  - The plastic or product shall have concentrations of regulated metals less than 50 % of those prescribed for sludge's or composts in the country where the product will be placed on the market or disposed of.

  - The germination rate and the plant biomass of the sample composts shall be no less than 90% that of the corresponding blank composts for two different plant species following OECD Guideline 208

Ultimate Aerobic Biodegradation as per ISO 14855:

Principle of Biodegradation:

Filobev / Filotub were submitted by ITC Limited Paperboards & Specialty Papers Division for testing under standard ISO/IS 14855-1. The test method determines the ultimate biodegradability and degree of disintegration of test material under conditions simulating an intensive aerobic composting process. During the aerobic biodegradation of the test material. Carbon dioxide. water, mineral salts, and new microbial cellular constituents (biomass) are the ultimate biodegradation products. The carbon dioxide produced is continuously monitored. or measured at regular intervals. in test and blank vessels to determine the cumulative carbon dioxide production. The percentage biodegradation is given by the ratio of the carbon dioxide produced from the test material to the maximum theoretical amount of carbon dioxide that can be produced from the test material. The maximum theoretical amount of carbon dioxide produced is calculated from the measured total organic carbon (TOC) content.

Compost Inoculum:

Well aerated compost from a property operating aerobic composting plant shall be used as the Inoculum. The inoculum shall be homogeneous and free from large inert objects such as glass, stones, or pieces of metal. Remove them manually and then sieve the compost on a screen of about 0.5cm to 1 cm. Determine the total dry solids and the volatile-solids content of the inoculum.

The total dry solids content shall be between 50 % and 55 % of the wet solids and the volatile solids no more than about 15 % of the wet or 30 % of the dry solids Adjust the water content, if necessary, before the compost is used by adding water or gentle drying, e.g., by aerating the compost with dry air. Prepare a mixture of 1 part of inoculum with 5 parts of deionized water MIX by shaking and measure the pH immediately, it shall be between 7,0 and 9,0. The compost inoculum should produce 50-150 mg of CO_2 per gram of volatile solids over the first 10 days of the test and an ash content of less than 70% and a pH between 7 and 8.2 is desired. The amount of total dry solids may range from 50 to 55%.

Figure 2: compost inoculum

Carbon Dioxide Analysis:

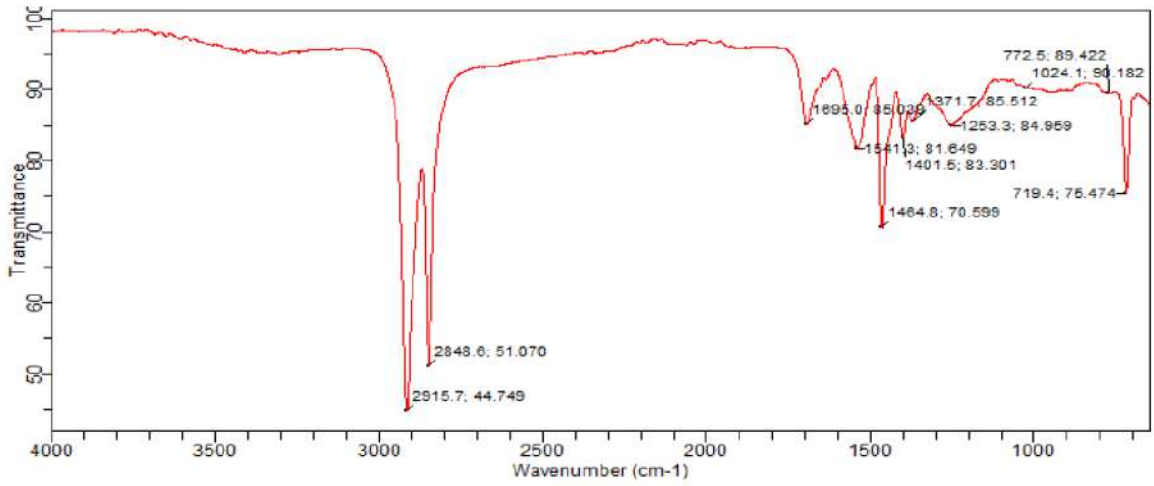
The carbon dioxide (CO_2) produced in each vessel reacted with $Ba(OH)_2$ and will be precipitated as barium carbonate ($BaCO_3$).The amount of carbon dioxide produced will be determined by titrating the remaining barium hydroxide with 0.05 N hydrochloric acid to a phenolphthalein end point. Data obtained from the titration will be used to calculate the amount of CO_2 produced.

RESULTS:

Chemical Characterization:

Table 1: Heavy Metal and volatile solid concentration in sample

Heavy Metals	Unit	Limits	Concentration
Volatile solids	%	Min 50	39.60
Zinc	mg/kg	150	1.45
Copper		50	1.07
Nickel		25	<0.10
Lead		50	<0.10
Mercury		0.5	<0.10
Chromium		50	1.42
Molybdenum		1	<0.10
Arsenic		0.75	<0.10
Selenium		5	<0.10
Cadmium		100	<0.10



Graph 1: FTIR graph of the Filobev / Filotub

The heavy metals and other toxic and hazardous waste are well within the précised limits of EN 13432 standards. FTIR spectra of **Filobev / Filotub** shown in **Graph 1** shows the IR bands characteristics.

Disintegration during Composting:

After 12 weeks, **Filobev / Filotub** sample started to disintegrate. After passing through 2mm sieve, the degree of disintegration of the test material **Filobev / Filotub** was found to be 98.0 %. Only 2 % was retained on the 2mm sieve.

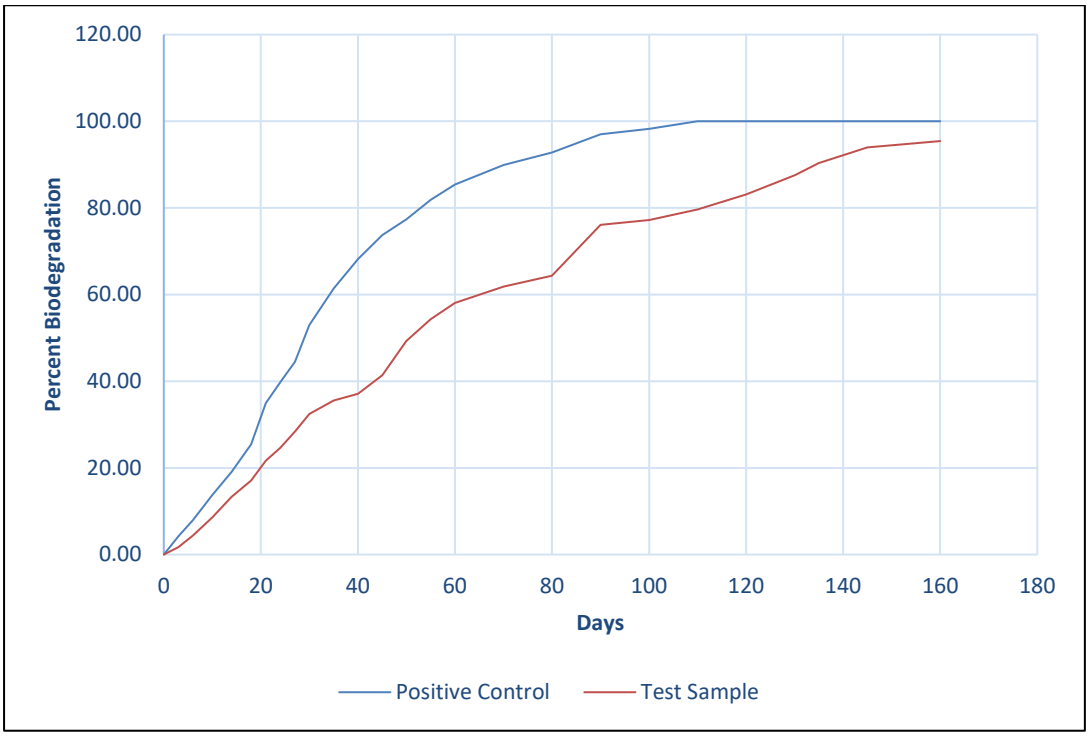
Ultimate Aerobic Biodegradation:

The **Filobev / Filotub** were subjected to biodegradation as per ISO 14855-1: biodegradability under controlled composting conditions at 58°C ± 2 °C and biodegradability was determined by measuring the actual metabolic conversion of the compostable material into carbon dioxide using the standard test method.

Table 2: Percentage Biodegradation of Positive control and Test samples

Day	% Biodegradation	
	Positive Control	Test samples
0	0.00	0.00
3	4.24	1.73
6	7.95	4.39
10	13.78	8.63
14	19.08	13.33
18	25.44	17.10
21	34.98	21.65
24	39.75	24.63
27	44.52	28.40
30	53.00	32.47
35	61.38	35.53
40	68.21	37.07
45	73.74	41.42
50	77.35	49.26
55	81.87	54.28
60	85.37	58.05
70	89.93	61.81
80	92.75	64.32
90	96.99	76.09
100	98.23	77.19
110	100.00	79.63
120	-	83.15
130	-	87.54
135	-	90.36
145	-	93.96
160	-	95.43

After 160 days of incubation under dry (58 °C ± 2 °C), aerobic controlled compositing conditions using test method ISO 14855-1, the reference (Positive control), and **Filobev / Filotub sample** were gradually biodegraded. The reference sample was degraded more than 100 % after 160 days while the **Filobev / Filotub sample** showed only 95.43 % after 160 days.



Graph 2: Percentage biodegradation of Filobev / Filotub Sample under aerobic composting

Quality of Compost:

Metal analysis of compost of test sample was done to check the toxic levels after biodegradation (Table 3). Seeds grown in compost prepared from samples showed more than 90% germination rate as compared to control. Root and shoot length of plant was similar or slightly more than controls (Table 4). There was no visual injury found in the roots and shoot of the plant due to the test substance.

Table 3: Analysis of compost residue collected after biodegradation.

PARAMETERS	UNIT	Test sample
pH	-	7.28
Mg	PPM	9953
Ca	PPM	26190
P	PPM	4105
Mn	PPM	833.9
Cu	PPM	114.1
Zn	PPM	250.6
Ni	PPM	48.35
Cd	PPM	<1.00
Pb	PPM	50.52
Hg	PPM	<1.00
Se	PPM	<1.00
As	PPM	<1.00
K	PPM	6053
Volatile solids	%	42.38
Dry Solids	%	42.67
Total Nitrogen	%	0.76

Impact of compost on plant growth:

Table 4: Germination Rate and Biomass of Brassica Juncea and Triticum aestivum seeds after 21 days.

	Plant species	Dose	Germination Rate (%)	Shoot Length (cm)	Root Length (cm)
Control	*Brassica juncea*	25%	99	12	10
		50%	98	10	10
	Triticum aestivum	25%	98	20	14
		50%	96	20	16

	Plant species	Dose	Germination Rate (%)	Shoot Length (cm)	Root Length (cm)
Test Sample	*Brassica juncea*	25%	98	12	10
		50%	95	12	12
	Triticum aestivum	25%	96	20	16
		50%	94	22	18

The above study was conducted at 25˚C ± 4 ˚C temperature, 66% ± 10% humidity and 54.07 FC light intensity with 16 hours of light. The above results showed that **Filobev / Filotub** Sample has no effect on the plant growth as well as no visible damage to the plants.

	
Control 25%	**Control 50%**
	
Test 25%	**Test 50%**

Figure 5: Effect of compost Filobev / Filotub *sample on Triticum aestivum* growth

	
Control 25%	**Control 50%**
	
Test 25%	**Test 50%**

Figure 6: Effect of compost Filobev / Filotub *sample on Brassica Juncea* growth

CONCLUSION:

In accordance to the conditions set forth in EN 13432, **Filobev / Filotub** samples submitted by **ITC LIMITED PAPERBOARDS & SPECIALTY PAPERS DIVISION** showed 95.43 % conversion of organic carbon to carbon dioxide after 160 days relative to the positive control. Filobev / Filotub sample disintegrated and after passing through 2mm sieve, the degree of disintegration of the test material was found to be 98.00 % after 12 weeks. Toxicity test was conducted as per OECD 208 on compost containing **Filobev / Filotub** sample residue. The compost sample did not show any effect on the seedling emergence, seedling growth and heavy metal toxicity test.

---------- **End of Report** ----------

Authorized Signatory

Jayashree Acharya
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