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(54) Title: BIODEGRADABLE PVC FILM FOR PHARMACEUTICAL PACKAGING AND A PROCESS FOR ITS PREPARATION

(57) Abstract: The present disclosure relates to a process for preparing a bio-degradable PVC based pharmaceutical grade thermo-formable film. The said film is stable in aerobic conditions and is bio-degradable under anaerobic conditions. The bio-degradable PVC based pharmaceutical grade film has application in the blister packing of pharmaceutical formulations.



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BIODEGRADABLE PVC FILM FOR PHARMACEUTICAL PACKAGING AND A PROCESS FOR ITS PREPARATION

FIELD OF THE DISCLOSURE:

The present disclosure relates to an eco-friendly PVC film and a container prepared there from for the blister packing of pharmaceutical formulations. The present disclosure also relates to a process for the preparation of the eco-friendly film.

BACKGROUND:

Blister packaging is a popular packaging method for pharmaceutical solid dosage forms which is growing rapidly. PVC based films are commonly used for this purpose as they possess suitable properties for thermo formation and protection. However, PVC being difficult to decompose, there has been request for degradable material from the industry.

There have been developments of various eco-friendly and biodegradable films, however, till date a biodegradable PVC film has not been commercially available.

Also, non PVC based materials lack the required thermal and chemical stability desirable for the manufacture of blister containers for pharmaceutical use.

Furthermore, the biodegradable material attempted to be developed in the prior art is susceptible to microbial growth at standard conditions. Presence of these types of material not only attracts microorganism, but also

adversely affects the physical properties and thermal/ chemical stability required for this application.

It is because of this unique consideration that is specifically applicable in the realm of pharmaceutical packaging, standard method of producing a biodegradable or pseudo biodegradable film, by having certain starch/ cellulose based polymers like polylactic acid or PVA or any such system in the formulation can not be applied as such for the preparation of blister containers for pharmaceutical formulations.

Recently, formulations for preparing biodegradable and compostable PVC which comprise pro-degradents have been disclosed in PCT Applications WO 2006/080955 and WO 2008/140552. The pro-degradents taught in these Applications comprise an adduct of an organotitanate or organozirconate or sulfonate with a hydrocarbon radical.

Though biodegradable and compostable films have been suggested in the prior art, the films are suitable only for the production of general purpose articles in the form of sheets for use in indoor and outdoor signs, bill boards, backdrops and wall coverings. The material of the prior art, however, suffers from many shortcomings and as such they are not suitable for the manufacture of pharma-grade blister packing materials. The material as suggested in the prior art has inherent processability limitations and it can not be subjected to the rigors of a calendering process for preparation of PVC film.

There is, therefore, felt a need for a process for preparation of a biodegradable and compostable PVC film specifically adapted for the manufacture of blister containers for pharmaceutical formulations.

The present disclosure is particularly directed to overcome the shortcomings associated with the disclosures in the prior art.

Definitions:

As used in the present disclosure, the following words and phrases are generally intended to have the meanings as set forth below, except to the extent that the context in which they are used to indicate otherwise.

The expression "pharmaceutical grade PVC" as used in the present disclosure, means a PVC material wherein the Vinyl Chloride Monomer content in said material is below 1 PPM, non toxic and complies to regulatory requirements for food and drug contact applications.

OBJECTS:

Some of the non-limiting objects which at least one embodiment of this disclosure may achieve are:

It is an object of the present disclosure to provide a process for preparation of a bio-degradable PVC film.

It is another object of the present disclosure to provide a bio-degradable PVC film for pharmaceutical applications.

It is another object of the present disclosure to provide a bio-degradable PVC film which is rigid.

It is still another object of the present disclosure to provide a bio-degradable PVC film which is not susceptible to the attack of microorganism in normal aerobic conditions.

It is still another object of the present disclosure to provide a bio-degradable PVC film which is capable of undergoing biodegradation under anaerobic conditions.

It is still another object of the present disclosure to provide a bio-degradable PVC film which is robust to withstand the environmental and mechanical stress which the film can experience during all stages of its processing.

It is another object of this disclosure to suggest a process for the manufacture of biodegradable PVC film and blister packs for pharmaceutical use made from these packs.

These and other objects of the present disclosure are to a great extent dealt in the disclosure.

SUMMARY:

In accordance with one aspect of the present disclosure there is provided a process for preparing a bio-degradable PVC based pharmaceutical grade thermo-formable film, said process comprising the following steps:

- a. mixing pharmaceutical grade PVC resin, at least one copolymer, at least one impact modifier, bio pro-degradant, at least one processing aid, and at least one stabilizer in a mixer to obtain a mixed batch of ingredients;

- b. extruding the mixed batch of ingredients in an extruder at a screw speed ranging between 2 rpm and 15 rpm and temperature ranging between 55 °C and 70 °C to obtain fluxed polymeric flakes; and
- c. calendering the polymeric flakes by subjecting them to at least two calender rolls maintained at temperatures ranging between 100 °C and 250 °C to obtain a bio-degradable PVC based pharmaceutical grade thermo-formable film,

wherein, said film is stable in aerobic conditions and is bio-degradable under anaerobic conditions.

Typically, the method step of mixing further comprises adding at least one pigment in the mixed batch.

Typically, the method step of mixing further comprises adding titanium dioxide in the mixed batch.

Typically, the method step of extruding comprises providing a difference between the torque of the feeder screw and torque of the output screw of the extruder which causes generation of pressure and heat on the mixture, resulting in fluxing of the material fed to the extruder.

Typically, the percentage difference between the torque of the feeder screw and torque of the output screw ranges between 5 and 20.

Preferably, the percentage difference between the torque of the feeder screw and torque of the output screw ranges between 8 and 16.

Typically, the pharmaceutical grade PVC resin is at least one selected from the group consisting of PVC suspension resin and PVC homopolymer suspension resin.

Typically, the copolymer is Vinyl Chloride/Vinyl Acetate copolymer.

Typically, the impact modifier is at least one selected from the group consisting of methylmethacrylate-butadiene-styrene-acrylic copolymer and acrylic modifier.

Typically, the bio pro-degradent is Ethylene-Vinyl Acetate copolymer with organoleptic additives.

Typically, the amount of bio pro-degradent ranges between 0.01 % and 20 % with respect to the mass of the film, preferably, 0.1 % and 10.0 % with respect to the mass of the film.

Typically, the processing aid is at least one selected from the group consisting of anti-blocking/slipping agents, antistatic agents, lubricants, release agents, anti-sticking agents and melt strength/viscosity balancing agents.

Typically, the stabilizer is at least one selected from the group consisting of polymer and soyabean stabilizer.

Typically, the at least two calender rolls are arranged at a distance ranging between 0.01 mm and 50 mm from each other.

Typically, the calender rolls are arranged in a cross-axial or bending position with respect to each other.

In accordance with another aspect of the present disclosure there is provided a bio-degradable PVC based pharmaceutical grade thermo-formable film obtained by a process of the present disclosure, said film comprising:

- i. a pharmaceutically grade PVC resin, ii. a copolymer, iii. at least one impact modifier, iv. a bio pro-degradant, v. at least one processing aid, vi. optionally, a titanium dioxide, vii. at least one stabilizer and viii optionally, at least one pigment,
- wherein, said film is stable in aerobic conditions and is bio-degradable under anaerobic conditions.

Typically, the pharmaceutical grade PVC resin is at least one selected from the group consisting of PVC suspension resin and PVC homopolymer suspension resin.

Typically, the copolymer is Vinyl Chloride/Vinyl Acetate copolymer.

Typically, the impact modifier is at least one selected from the group consisting of methylmethacrylate-butadiene-styrene-acrylic copolymer and acrylic modifier.

Typically, the bio pro-degradant is Ethylene-Vinyl Acetate copolymer with organoleptic additives.

Typically, the amount of bio pro-degradant ranges between 0.01 % and 20 % with respect to the mass of the film, preferably, 0.1 % and 10.0 % with respect to the mass of the film.

Typically, the processing aid is at least one selected from the group consisting of anti-blocking/slipping agents, antistatic agents, lubricants, release agents, anti-sticking agents and melt strength/viscosity balancing agents.

Typically, the stabilizer is at least one selected from the group consisting of polymer and soyabean stabilizer.

Typically, the PVC film is rigid.

In accordance with another aspect of the invention there is provided a blister pack made from the PVC film.

Brief description of the accompanying drawings:

Figure 1- illustrates the process for preparing the bio-degradable PVC based pharmaceutical grade thermo-formable film of the present disclosure,

wherein, a= mixing and fluxing unit, b= conveyor belt unit, c= calender rolls, d= post calender unit, and e= winder unit.

Figure 2- illustrates the transmission rate data graph of the bio-degradable PVC film prepared in Example 1,

Figure 3- illustrates the FTIR graph of side A of the bio-degradable PVC film prepared in Example 1,

Figure 4- illustrates the FTIR graph of side B of the bio-degradable PVC film prepared in Example 1,

Figure 5- illustrates the heat seal strength of the bio-degradable PVC film prepared in Example 1, and

Figure 6- illustrates the tensile strength of the bio-degradable PVC film prepared in Example 1.

DETAILED DESCRIPTION:

In accordance with one aspect of the present disclosure there is provided a process for preparing a bio-degradable and compostable PVC based pharmaceutical grade thermo-formable film specifically suitable for the manufacture of blister container for pharmaceutical formulations.

The process for preparing a biodegradable and compostable PVC film is specifically adapted for pharmaceutical applications, especially the preparation of blister containers in accordance with the present disclosure.

In the mixing step, the ingredients comprising PVC resin, copolymer, at least one impact modifier, bio pro-degradant, at least one processing aid, at least one stabilizer and optionally, titanium dioxide is added to a mixer and mixed thoroughly to ensure that all the ingredients are mixed uniformly prior to feeding into the extruder. Further, at least one pigment may be added depending upon the need and requirement during or after the mixing step. The pharmaceutical grade PVC resin is at least one selected from the group consisting of PVC suspension resin and PVC homopolymer suspension resin.

The copolymer may be Vinyl Chloride/Vinyl Acetate copolymer.

Further, the impact modifier employed is at least one selected from the group consisting of methylmethacrylate-butadiene-styrene-acrylic copolymer and acrylic modifier.

The stabilizer in accordance with the present disclosure is at least one selected from the group consisting of a polymer and soyabean stabilizer.

The major equipment employed for carrying out this method step includes but is not limited to turbo (heated) mixer and cooling mixer.

The mixed batch is continuously fed into an extruder. The extruder produces fluxed material which is as a result of the conversion of the powdered batch into fluid material. The extruder creates the fluxed material with a minimum of entrapped air but without overheating or overworking the material.

The method step of extrusion is carried out in a kneader, typically a ko-kneader (KK) which has three primary process adjustments i.e., power feeder torque, screw speed and temperature. The power feeder is a hopper with an internal auger mounted atop the KK which supplies powdered blend to the KK. Increase in the power feeder speed forces more blend into the KK, which increases the torque. Further, changing the speed of the KK screw changes the rate of output. The percentage difference between the torque of the feeder screw and the torque of the output screw causes generation of pressure and heat on the mixture and therefore fluxing of the

material which comes out in the form of flakes to be fed to the calender. The KK output is matched with the calender input to maintain a consistent level. The KK has three temperature control zones. The temperature of each zone affects the fluxing of the powdered blend and different ingredients require different temperature settings.

The bio pro-degradant typically employed for the preparation of the PVC film of the present disclosure comprises Ethylene-Vinyl Acetate copolymer with organoleptic additives. The presence of the bio pro-degradant adversely affects the gelification process of the composite and makes extrusion process very difficult. In accordance with the process of the present disclosure, the gelification of the composition is carried out by controlling specific processing parameters during extruding. These parameters include power torque, speed and temperature. The high torque ensures the desired level of gelification whereas the optimum temperature range during the process of the present disclosure is such that it does not allow the film to become hazy. The mixed batch is fluxed by applying power feeder torque difference ranging between 5% and 20%, screw speed ranging between 2 rpm and 15 rpm and temperature ranging between 55 °C and 70 °C to obtain flakes. In accordance with the process of the present disclosure, the torque difference between the feeder screw and the output screw, particularly, ranges between 8% and 16%. In accordance with an exemplary embodiment of the present disclosure if the input feeder power is "x" then the power of the output and therefore the speed ranges between $(95/100)x$ and $(80/100)x$

In accordance with the process of the present disclosure, the temperature of the mixture is lowered by 3 °C to 5 °C in order to avoid any degradation of the additive.

The extruded polymeric flakes are then made to fall on heated calender rolls where it melt and forms a film. Calendering converts the fluxed material into films of required thickness by passing through multiple heated and cooled rolls. The calender rolls have three primary means of adjustment i.e., temperature, gap and speed. In addition, cross-axis or roll bending adjustments may also be used to fine-tune the film profile. Cross-axis and roll bending offset the characteristic “oxbow” profile associated with calendered film.

Temperature parameters of calender rolls affect the viscosity of the material, which relates to behavior in the banks and overall surface quality. Differences in temperature from roll to roll may be used to facilitate the transfer of material from one roll to the next. Too high a temperature may cause degradation (discoloration, decomposition) of the material as well as a tendency to stick to the calender rolls whereas too low temperature may result in higher air entrapment, more visible flow lines, and overall poor surface quality. Further, high temperature makes the composition rigid and affects the calendering process where the polymer particles get burnt resulting in the formation of black particles. This rigidity also creates non-uniformity of the calendered films and affects thickness uniformity which is essential for blister packing application. This makes the film unsuitable for pharmaceutical applications, and especially, for the preparation of blister containers for pharmaceutical formulations.

The inventors of the present disclosure have employed processing aids to nullify the rigidity effect due to the presence of bio pro-degradant additives, and thus reduce the rigidity of the films. The additives as employed in accordance with the process of the present disclosure also ensure uniformity of the film. Furthermore, these additives obviate the possibility of the formation of black particles during processing and thereby ensuring the preparation of films that are suitable for pharmaceutical applications, especially, for the preparation of blister containers for pharmaceutical formulations. Further, to achieve optimum quality film the temperature of the calender rolls is maintained between 100 °C and 250 °C as the take-off and cooling roll temperatures affect shrinkage characteristics and final thickness profile.

The thickness of the film depends on the gap between the two calender rolls. The gap between the two calender roll ranges between 0.01 mm and 50 mm.

Rotating speeds of the calender rolls affect overall line throughput and surface quality due to residence time on the calender rolls.

The post-calender section is adjustable for temperature and speed. The goal of both calender and post-calender controls is to produce film of uniform thickness with minimal surface imperfections and acceptable shrinkage characteristics.

The film obtained by the process of the present disclosure is stable in aerobic conditions whereas bio-degradable under anaerobic conditions.

The film obtained is then cooled prior to winding into roll form by employing a winder. The equipment employed for carrying out this process of extrusion and calendering includes but are not limited to extruder (kneader), calender rolls (heated), post calender rolls (heat & cold) and winders.

In another aspect of the present disclosure there is provided a bio-degradable PVC based pharmaceutical grade thermo-formable film prepared in accordance with the process of the present disclosure.

The PVC film envisaged in accordance with the present disclosure is compostable and anaerobically biodegradable under a landfill. Further, the PVC film of the present disclosure is also made in the form of a “paper lookalike” feel, texture and appearance.

In accordance with one of the embodiment of the present disclosure there is provided a generally pharmaceutical grade thermoforming PVC film composition comprising: i) PVC resin; ii) copolymer; iii) at least impact modifier iv) bio pro-degradant; v) at least one processing aid; vi) optionally, titanium dioxide and vii) at least one stabilizers and viii) optionally, at least one pigment. The film of the present disclosure is stable in aerobic conditions and is bio-degradable under anaerobic conditions.

The bio pro-degradant employed for the preparation of the PVC film of the present disclosure includes but is not limited to Ethylene-Vinyl Acetate copolymer with organoleptic additives. The presence of bio pro-degradant in specific proportions renders the PVC film of the present disclosure

compliant for making blister container for pharmaceutical formulations while retaining its biodegradability characteristics under anaerobic conditions. The amount of the bio pro-degradant in the PVC film of the present disclosure ranges between 0.01% to 20%, preferably between 0.1% to 10% with respect to the mass of the film. The bio pro-degradant helps microbes to break down the long polymer molecule under anaerobic conditions, especially under the ground and mineralize it into CO₂, methane, water and biomass.

The processing aid in accordance with the present disclosure is at least one selected from the group consisting of anti-blocking/slipping agents, antistatic agents, lubricants, release agents, anti-sticking agents and melt strength/viscosity balancing agents.

The PVC resin used for making of the film of the present disclosure is specifically devoid of any plasticizer as plasticizers tend to leach/migrate to the substance in contact. Therefore, as per the regulatory requirement, pharmaceutical grade PVC film has to be a rigid plasticizer free film.

Mobility in the polymer matrix is essential for biodegradability. The PVC film of the present disclosure comprises a polymer system which ensures internal mobility in the polymer matrix and allows the functioning of the bio pro-degrade system without the use of a plasticizer.

In accordance with yet another aspect of the present disclosure there is provided a complete biodegradable blister container prepared from the PVC

film of the present disclosure as the cavity forming material and a lidding foil which is a paper based.

The disclosure will now be described with the help of following non-limiting examples.

Examples:

Example I: Preparation of biodegradable and compostable white opaque PVC film of 250 micron

A. Batch Mixing Operation:

255.7 kg of PVC suspension resin, 49.90 kg of Vinyl chloride/vinyl Acetate Copolymer, 14.52 kg of Methylmethacrylate-Butadiene- Styrene Terpolymer, 39.50 kg of PVC emulsion polymer, 1.50 kg of polyol ester based lubricant, 0.5 kg of Amide of ethylenediamine, 2.05 kg of Polyvinyl chloride, 2.06 kg of Acrylic polymer processing aid, 2.42 kg of butadiene/methylmethacrylate/styrene, 4.00 kg of Bio Pro-degradent (Eco-pureTM manufactured by Bio-tech Environmental, LLC), 11.40 kg of Titanium dioxide, 3.75 kg of Polymer stabilizer and 3.04 kg of Partial esters of fatty acids with glycerol were added to the batch mixing system from the respective storage system using programmable logic controlled material dosage/discharge systems and mixed to obtain a thoroughly mixed batch of ingredients.

B. Calender Operation

The following process protocol was followed for carrying out the calendering operation.

PROCESS STEP	EQUIPMENT	PARAMETER		UNIT	VALUE
Extruder	Ko-Kneader	Difference between the torque of the feeder screw and the torque of the output screw		%	10.6
		Screw speed		rpm	7.6
Calendering		Temperature	Cylinder 1	°C	166
		Speed		m/min	15.4
		Temperature	Cylinder 2	°C	168
		Speed		m/min	16.5
		Temperature	Cylinder 3	°C	175
		Speed		m/min	17.7
		Temperature	Cylinder 4	°C	178
		Speed		m/min	19.4
		Temperature	Cylinder 5	°C	154
		Speed		m/min	23.0
Winder		Web tension		(dN)	325

The process is followed as below

- Set the power, speed of the KK screw and the temperature of the three zones to the required level.
- Continuously feed the mixture of ingredients produced at the batch mixing operation to Ko-Kneader.

- Fine tune the power feeder torque difference, speed and temperature of KK as per the process protocol so that flakes come out uniformly.
- Set the gap, speed, and temperature of the calender rolls to the required level as per the process protocol. The parameter setting should also consider thickness, gloss, clarity and surface roughness requirement.
- Start feeding the fluxed materials to the calender rolls.
- Fine tune the parameters, gap, speed and the temperature of each calender roll as per the process protocol so that the film with required thickness, clarity and gloss comes out at the end.
- Rewind the formed film in to rolls with the specified tension .

The Calender roll gaps provided the initial thickness control of the material and final thickness was determined by draw at the take-off rolls.

C. Post Calender operations.

These master rolls further can be slit into small rolls .

D. TEST DATA

The Bio degradable film thus produced is tested for following properties to prove its application for blister packing application.

1. The physical and thermo mechanical properties for the application of blister packing,
2. Blister forming machine trials,
3. Food and drug contact application compliances, and
4. Biodegradability tests.

RESULTS:

1. The physical and thermo mechanical properties for the application of blister packing

Sr. No	Parameters	Test Method	Unit	250 Micron Bio-PVC film as prepared in Example I	Specification of General Bilcare 250 Micron PVC film
1	Total Thickness	DIN 53370	Micron	249	250 ± 5 %
2	Total GSM	DIN EN ISO 2286	GSM	346	345 ± 5 %
3	Impact Strength	ASTM D1709	gm	1290	350 min.
4	Specific Gravity Opaque	In-house	gm/cc	1.38	1.38 ± 10 %
5	Average Yield Opaque	In-house	m ² /kg	2.90	2.90
6	Tensile Strength	ASTM D882	Kg/cm ²		
	Longitudinal			457.92	400 min
	Transverse			540.92	400 min
7	Peak Elongation	ASTM D882	%		
	Longitudinal			3.62	4 Min
	Transverse			3.79	4 Min
8	Dimensional Stability	ASTM D1204	%		
	Longitudinal			-9	-7 max
	Transverse			+2	+ 2max
9	Heat Seal Strength(PVC to Al. foil)	In-house	Kgf/cm	0.774	0.30 min

The physical and thermo mechanical properties of the bio-degradable PVC film prepared in accordance with the process of the present disclosure are at par with the non-degradable 250 microns PVC film.

2. Blister forming machine trials

Thermoforming trials were taken on rotary vacuum forming as well flat pressure forming thermoforming machines with various cavity sizes and found that it perform exactly the same as the regular thermoforming PVC films. The thermoforming parameters remain the same with that of regular films.

Sr. No	Parameters	Test Method	Unit	250 Micron Bio-PVC film as prepared in Example I	Thermoforming temperature of General Bilcare 250 Micron PVC film
1	Thermo formability in rotary vacuum forming machine at 20-25 Inches of vacuum	In-house	$^{\circ}\text{C}$	155- 158 $^{\circ}\text{C}$	155- 158 $^{\circ}\text{C}$
2	Thermo formability in flat bed pressure forming machine at 5-6 Kg pressure	In-house	$^{\circ}\text{C}$	110- 125 $^{\circ}\text{C}$	110- 125 $^{\circ}\text{C}$

Thermo-formability of the bio-degradable PVC film prepared in accordance with the process of the present disclosure is at par with the non-degradable 250 microns PVC film.

3. Food and drug contact material compliances

Sr. No	Parameters	Test Method	Unit	250 Micron Bio-PVC film as prepared in Example I	Specification of General Bilcare 250 Micron PVC film
1	Toxicity Test	USP Current Ed.		Non-toxic	Non-toxic
2	VCM content	2002/72/EC	ppm	<1	<1
3	Global migration	2002/72/EC	ppm	5.1	<60
4	Average WVTR @ 90%RH at 38°C	ASTM F1249	grams/m ² /24 hr.	3.72	
5	Average OTR @ 0%RH at 23°C	ASTM D3985	cc/m ² /24 hr.	17.322	
6	FTIR	In-house	-	Complies	Complies
7	Heavy Metal Analysis				
A	Cadmium	ICP-OES/AES	ppm	< 1	< 1
B	Barium			< 1	< 1
C	Iron			2	2
D	Lead			< 1	< 1
E	Arsenic ppm			< 1	< 1
F	Antimony ppm			< 1	< 1
G	Chromium			< 1	< 1

H	Mercury			< 1	< 1
I	Tin			10	10
J	Zinc			1	1

Food and drug contact material compliances of the bio-degradable PVC film prepared in accordance with the process of the present disclosure is at par with the non-degradable 250 microns PVC film.

4. Biodegradability tests:

Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions

Inoculum Source:

- Organic Compost – McEnroe Organic Farms, Millerton, NY
Mattabassit Waste Treatment Facility Anaerobic
Digestion
 - ❖ Solid Content 22%
 - ❖ pH 8.2
 - ❖ Volatile Fatty Acids 0.7 g/kg
 - ❖ Ammonia Nitrogen 1.0 mg/kg
 - ❖ Volatile Solids 24.9 %

Procedure:

1. Three weighed replicates of the test material were prepared by placing them into 1000 grams of inoculum in containers which were then attached to the gas measuring devices. Incubation temperatures of $52 \pm 2^{\circ}\text{C}$ were maintained by placing the containers in temperature controlled incubators.
2. Three blanks containing only inoculum, were prepared as described in (1) above, as were three positive controls each containing 20 grams of

thin layer grade cellulose. Three negative controls were also run utilizing untreated samples supplied by Northeast Laboratories.

3. Samples were incubated for forty five days in the dark, or at times, diffused light. Gas volumes were determined daily. Carbon Dioxide and Methane concentration were also determined. Temperature and room atmospheric pressures were monitored during the course of incubation.

The results are shown in the following tables.

Gas Production Data – Samples

Day	250 Micron Bio-PVC film as prepared in Example I			Negative Control PE			Positive Control Cellulose			Inoculum Control	
	A	B	C	A	B	C	A	B	C	A	B
Totals Days 1-30	9473	10659	9877	3609	4435	3992	15662	18075	16348	3867	3180
31	25	25	50	155	180	150	185	150	205	40	50
32	25	25	50	155	180	150	185	150	205	40	50
33	42	106	85	32	106	74	53	106	32	32	42
34	116	129	110	16	97	48	113	90	90	32	32
35	116	129	110	16	97	48	113	90	90	32	32
36	116	129	110	16	97	48	113	90	90	32	32
37	274	274	205	34	71	68	205	171	171	102	102
38	274	274	205	34	71	68	205	171	171	102	102
39	274	274	205	34	71	68	205	171	171	102	102
40	40	50	200	50	50	50	100	100	130	70	80
41	51	51	44	17	13	27	68	68	68	137	68
42	51	51	44	17	13	27	68	68	68	137	68
43	51	51	44	17	13	27	68	68	68	137	68
44	69	87	52	17	0	24	52	52	62	0	13
45	69	87	52	17	0	24	52	52	62	0	13
46	0	10	0	50	20	50	50	50	30	30	10

47	9	9	9	28	15	38	67	57	67	28	19
Totals	11075	12420	11452	4314	5529	4981	17564	19779	18128	4920	4063
Averages	11649			4941			18490			4728	

Methane and Carbon Dioxide Readings

Day	250 Micron Bio-PVC film as prepared in Example 1		Negative Control 100 % PE Plastic		Positive Control Cellulose		Inoculum Control	
% Average	Methane %	Carbon Dioxide %	Methane %	Carbon Dioxide %	Methane %	Carbon Dioxide %	Carbon Dioxide %	Methane %
Averages Days 1-30	25.7 %	22.2 %	20.5 %	17.6 %	55 %	35.2 %	25.2 %	23 %
34	26 %	18 %	18.4 %	10.2 %	49 %	38.4 %	22 %	15.7 %
38	25 %	15 %	13.1 %	11.6 %	51 %	39.1 %	24 %	13.6 %
43	25 %	17 %	17.4 %	11.2 %	48 %	32.3 %	20 %	13.5 %
Average	25.6 %	20.4 %	24.9 %	15.4 %	53.1 %	35.7 %	24.1 %	20.1 %

Calculations of Results

Sample	Average Weight grams	Average Gas Vol. (mL)	Methane			Carbon Dioxide			Total CH ₄ + CO ₂	Sample-Inoculum=
			(%)	(mL)	(Wt) C (grams)	(%)	(mL)	(Wt) C (grams)		
250 Micron Bio-PVC film as prepared in Example 1	25	11649	25.6	2982	1.6	20.4	2376	1.3	2.9	1.8

0.4	1.1	0
3.5	8.8	7.7
0.5	1.1	

(%)
Biodegradation
Days 1-45
18.7 %
0 %
87.5 %

7 % biodegradation

511-02. The results

eking application. It

stable Glass Clear

Methylmethacrylate-

ple Pressed Stearic

0 kg of Soyabean

stabilizer, 4.910 kg of Amide of Ethylenediamine, 4.420 kg of Polyvinyl chloride, 1.970 kg of Acrylic polymer processing aid, 3.440 kg of Methylmethacrylate-Butadiene-Styrene processing aid, 3.440 kg of Bio Pro-degradant (Eco-pureTM manufactured by Bio-tech Environmental, LLC), 1.180 kg of Fatty acid ester of poly functional alcohols and 4.420 kg of Polymer stabilizer were added to the batch mixing system from the respective storage system using programmable logic controlled material dosage/discharge systems and mixed to obtain a thoroughly mixed batch of ingredients.

B. Calender Operation:

The following process protocol as developed by the inventors of the present disclosure was followed for carrying out the calendering operation.

PROCESS STEP	EQUIPMENT	PARAMETER		UNIT	VALUES
Extruder	Ko-Kneader	Difference between the torque of the feeder screw and the torque of the output screw		%	10.19
		Screw speed		Rpm	17.6
Calendering		Temperature	Cylinder 1	o C	174
		Speed		m/min	20.3
		Temperature	Cylinder 2	o C	177
		Speed		m/min	22.3
		Temperature	Cylinder	o C	196

	Speed	3	m/min	24.7
	Temperature	Cylinder 4	o C	205
	Speed		m/min	28.1
	Temperature	Cylinder 5	o C	162
	Speed		m/min	33.5
Winder	Web tension		(dN)	350

The film was prepared by the process as described in Example I.

C. TEST DATA

The Bio degradable film thus produced is tested for following properties to prove its application for blister packing application.

1. The physical and thermo mechanical properties for the application of blister packing,
2. Blister forming machine trials, and
3. Biodegradability tests.

RESULTS:

1. The physical and thermo mechanical properties for the application of blister packing

Sr. No	Parameters	Test Method	Unit	250 Micron Bio-PVC film as prepared in Example II	Specification of General Bilcare 250 Micron PVC film
1	Total Thickness	DIN 53370	Micron	253	250 ± 5 %
2	Total GSM	DIN EN ISO 2286	GSM	338	340 ± 5 %
3	Impact Strength	ASTM D1709	gm	1200	350 min.
4	Specific Gravity Opaque	In-house	gm/cc	1.35	1.35 ± 5 %
5	Average Yield Opaque	In-house	m ² /kg	2.96	2.90
6	Tensile Strength	ASTM D882	Kg/cm ²		
	Longitudinal			457.92	400 min
	Transverse			540.92	400 min
7	Peak Elongation	ASTM D882	%		
	Longitudinal			3.62	4 Min
	Transverse			3.79	4 Min
8	Dimensional Stability	ASTM D1204	%		
	Longitudinal			-7	-7 max
	Transverse			+ 2	+ 2max
9	Heat Seal Strength(PVC to Al. foil)	In-house	Kgf/cm	0.80	0.30 min

The Physical and thermo mechanical properties of the bio-degradable PVC film prepared in accordance with the process of the present disclosure are at par with the non-degradable 250 microns PVC film.

2. Blister forming machine trials

Thermoforming trials were taken on rotary vacuum forming as well flat pressure forming thermoforming machines with various cavity sizes and found that it perform exactly same as the regular thermoforming PVC films. The thermoforming parameters remain same with that of regular films.

Sr. No	Parameters	Test Method	Unit	250 Micron Bio-PVC film as prepared in Example II	Thermoforming temperature of General Bilcare 250 Micron PVC film
1	Thermo formability in rotary vacuum forming machine at 20-25 Inches of vacuum	In-house	$^{\circ}\text{C}$	150- 155 $^{\circ}\text{C}$	150- 155 C
2	Thermformability in flat bed pressure forming machine at 5-6 Kg pressure	In-house	$^{\circ}\text{C}$	105- 120 $^{\circ}\text{C}$	105- 120 $^{\circ}\text{C}$

Thermo formability of the bio-degradable PVC film prepared in accordance with the process of the present disclosure is at par with the non-degradable 250 microns PVC film.

3. Biodegradability tests:

Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions

Inoculum Source:

- Organic Compost – McEnroe Organic Farms, Millerton, NY
Mattabassit Waste Treatment Facility Anaerobic
Digestion
 - ❖ Solid Content 22%
 - ❖ pH 8.2
 - ❖ Volatile Fatty Acids 0.7 g/kg
 - ❖ Ammonia Nitrogen 1.0 mg/kg
 - ❖ Volatile Solids 24.9 %

Procedure: The procedure followed was the same as described in Example I

Theoretical Gas Production

Samples	Carbon Content (grams)	Methane (grams)	Carbon Dioxide (grams)
250 Micron Bio-PVC film as prepared in Example II	9.61	12.9	35.2
Negative Control (PE) (25 grams)	21.4	28.6	78.5
Positive Control (Cellulose) (20 grams)	8.8	11.8	32.3

Gas Production Data - Samples

Day	250 Micron Bio-PVC film as prepared in Example II			Negative Control PE			Positive Control Cellulose			Inoculum Control		
	A	B	C	A	B	C	A	B	C	A	B	C
Totals 1-30	4992	5012	5153	1657	1298	1296	19334	19621	20238	2597	2283	2142
Totals 31-45	738	621	562	309	311	271	1030	954	1137	420	313	366
Avg 31-45	640			297			1040			366		

Methane and Carbon Dioxide Readings

Day	250 Micron Bio-PVC film as prepared in Example II		Negative Control PE		Positive Control Cellulose		Inoculum Control	
%	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide
33	16 %	13.8 %	12 %	7.6 %	29 %	25.9 %	13 %	9.3 %
38	21 %	12.9 %	9 %	7.3 %	32 %	29.7 %	10 %	7.6 %
42	13 %	10.2 %	8 %	7.9 %	26 %	24.2 %	9 %	9.1 %
Avg	16.7 %	12.3 %	9.7 %	7.6 %	29 %	26.6 %	10.7 %	8.7 %

Calculations of Results

Sample	Average Weight grams	Average Gas Vol. (mL)	Methane			Carbon Dioxide			Total CH ₄ + CO ₂	Sample-Inoculum
			(%)	(mL)	(Wt) C (grams)	(%)	(mL)	(Wt) C (grams)		
250 Micron Bio-PVC film as prepared in Example II	25	640	16.7	107	0.057	12.3	79	0.042	0.099	0.061
Negative Control PE	25	297	9.7	29	0.016	7.6	23	0.012	0.028	0
Positive Control	20	1040	29	302	0.16	26.7	278	0.15	0.31	0.272
Inoculum Control	1000	366	10.7	39	0.021	8.7	32	0.017	0.038	

Results (Average of 3)				
	Gaseous Carbon Recovered	Theoretical Grams	(%) Biodegradation Days 31-45	(%) Biodegradation Days 1-45
250 Micron Bio-PVC film as prepared in Example II	0.06	9.61	0.065%	8.015 %
Negative Control PE	0	21.4	0 %	0 %
Positive Control	0.272	8.8	3.09 %	91.09 %

The results support the suitability of the product for the blister packing application. It also depicts the biodegradation of the film.

Example III: Preparation of biodegradable and compostable and stretched PVC film of 250 micron

A. Batch Mixing Operation:

187.00 kg of PVC homopolymer suspension resin, 243.00 kg of Vinyl chloride/Vinyl Acetate copolymer, 34.100 kg of Methylmethacrylate-Butadiene- Styrene acrylic copolymer, 6.330 kg of Polymer stabilizer, 21.900 kg of Dicarboxylic acid ester, 1.460 kg of Fatty acid ester of polyfunctional alcohols, 1.220 kg of butadiene/ methylmethacrylate /styrene processing aid, 0.487 kg of Montanic ester wax, 0.414 kg of Mg-Silicate based talc and 3.410 kg of Bio Pro-degradent (Eco-pureTM manufactured by Bio-tech Environmental, LLC) were added to the batch mixing system from

the respective storage system using programmable logic controlled material dosage/discharge systems and mixed to obtain a thoroughly mixed batch of ingredients.

B. Calender Operation:

The following process protocol was followed for carrying out the calendering operation.

PROCESS STEP	EQUIPMENT	PARAMETER		UNIT	VALUE
Extruder	Ko-Kneader	Difference between the torque of the feeder screw and the torque of the output screw		%	12.09
		Screw speed		rpm	9.4
Calendering		Temperature	Cylinder 1	°C	159
		Speed		m/min	14.6
		Temperature	Cylinder 2	°C	160
		Speed		m/min	16.1
		Temperature	Cylinder 3	°C	174
		Speed		m/min	17.8
		Temperature	Cylinder 4	°C	193
		Speed		m/min	20.7
		Temperature	Cylinder 5	°C	162
		Speed			25.2
Winder		Web tension		(dN)	250

The film was prepared by the process as described in Example I.

C. TEST DATA

The Bio degradable film thus produced is tested for Biodegradability properties to prove its application for blister packing application.

RESULTS:

1. Biodegradability tests:

Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions

Inoculum Source:

- Organic Compost – McEnroe Organic Farms, Millerton, NY
Mattabassit Waste Treatment Facility Anaerobic Digestion
 - ❖ Solid Content 22%
 - ❖ pH 8.2
 - ❖ Volatile Fatty Acids 0.7 g/kg
 - ❖ Ammonia Nitrogen 1.0 mg/kg
 - ❖ Volatile Solids 24.9 %

Procedure: The procedure followed was the same as described in Example I

Theoretical Gas Production

Samples	Carbon Content (grams)	Methane (grams)	Carbon Dioxide (grams)
250 Micron Bio-PVC film as prepared in Example III	9.61	12.9	35.2
Negative Control (PE) (25 grams)	21.4	28.6	78.5
Positive Control (Cellulose) (20 grams)	8.8	11.8	32.3

Gas Production Data - Samples

Day	250 Micron Bio-PVC film as prepared in Example III			9.61			12.9			35.2		
	A	B110	258C	A	B	C	A	B	C	A	B	C
Totals 1-30	4752	5203	4344	1657	1298	1296	19334	19621	20238	2557	2283	2142
Totals 31-45	748	716	800	311	271	1030	1030	954	1137	420	313	366
Avg 31-45	1085			297			1040			366		

Methane and Carbon Dioxide Readings

Day	250 Micron Bio-PVC film as prepared in Example III		9.61		12.9		35.2	
%	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide
33	15 %	13.8%	12%	7.6 %	29 %	25.9 %	13 %	9.3 %
38	15 %	10.9 %	9 %	7.3 %	26 %	24.2 %	10 %	7.6 %
42	19 %	12.1 %	8 %	7.9 %	26 %	24.2%	9 %	9.1 %
Avg	16.3 %	12.3 %	9.7 %	7.6 %	29 %	26.6 %	10.7%	8.7 %

Calculations of Results

Sample:	Average Weight grams	Average Gas Vol. (mL)	Methane			Carbon Dioxide			Total CH ₄ + CO ₂	Sample-Inoculum
			(%)	(mL)	(Wt) C (grams)	(%)	(mL)	(Wt) C (grams)		
250 Micron Bio-PVC film as prepared	25	755	16.3	123	0.066	12.3	93	0.05	0.116	0.078

in Example III										
Negative Control PE	25	297	9.7	29	0.016	7.6	23	0.012	0.028	0
Positive Control	20	1040	29	302	0.16	26.7	278	0.15	0.31	0.272
Inoculum Control	1000	366	107	39	0.028	8.7	32	0.017	0.038	

Results (Average of 3)				
	Gaseous Carbon Recovered	Theoretical Grams	(%) Biodegradation Days 31-45	(%) Biodegradation Days 1-45
250 Micron Bio-PVC film as prepared in Example III	0.078	9.61	0.81	7.2
Negative Control PE	0	21.4	0	0
Positive Control	0.272	8.8	3.09	91.09

The results support the suitability of the product for the blister packing application. It also depicts the biodegradation of the film prepared in accordance with the process of the present disclosure.

Example IV:

A. Batch Mixing Operation:

187.00 kg of PVC homopolymer suspension resin, 243.00 kg of Vinyl chloride/Vinyl Acetate copolymer, 34.100 kg of Methylmethacrylate-Butadiene- Styrene acrylic copolymer, 6.330 kg of Polymer stabilizer, 21.900 kg of Dicarboxylic acid ester, 1.460 kg of Fatty acid ester of polyfunctional alcohols, 1.220 kg of butadiene/ methylmethacrylate /styrene processing aid, 0.487 kg of Montanic ester wax, 0.414 kg of Mg-Silicate based talc and 3.410 kg of Bio Pro-degradent (Eco-pureTM manufactured by Bio-tech Environmental, LLC) were added to the batch mixing system from the respective storage system using programmable logic controlled material dosage/discharge systems and mixed to obtain a thoroughly mixed batch of ingredients.

B. Calender Operation:

The following process protocol as developed by the inventors of the present disclosure was followed for carrying out the calendering operation.

The same manufacturing process as in the example 1 is followed till the calendering stage. Further the film is heated and stretched in both directions to make it a thinner film. These films are used for shrink packaging applications.

PROCESS STEP	EQUIPMENT	PARAMETER		UNIT	VALUE
Extruder	Ko-Kneader	Difference between the torque of the feeder screw and the torque of the output		%	12.09

		screw			
		Screw speed		rpm	9.4
Calendering	Temperature	Cylinder 1	o C	159	
	Speed		m/min	14.6	
	Temperature	Cylinder 2	o C	160	
	Speed		m/min	16.1	
	Temperature	Cylinder 3	o C	174	
	Speed		m/min	17.8	
	Temperature	Cylinder 4	o C	193	
	Speed		m/min	20.7	
	Temperature	Cylinder 5	o C	162	
	Speed		m/min	25.2	
Winder	Web tension		(dN)	250	
Stenter	Temperature	Heat Zone 1	o C	105	
		Heat Zone 2	o C	95	
		Heat Zone 3	o C	90	
		Stretch Zone1	o C	88	
		Stretch Zone 2	o C	88	

C. Test Data

The film thus produced is tested for its biodegradability

Results:

Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions

Inoculum Source:

- Organic Compost – McEnroe Organic Farms, Millerton, NY
Mattabassit Waste Treatment Facility Anaerobic

Digestion

❖ Solid Content	22%
❖ pH	8.2
❖ Volatile Fatty Acids	0.7 g/kg
❖ Ammonia Nitrogen	1.0 mg/kg
❖ Volatile Solids	24.9 %

Procedure: The procedure followed was the same as described in Example I

The results are shown in the following tables.

Theoretical Gas Production

Samples	Carbon Content (grams)	Methane (grams)	Carbon Dioxide (grams)
Bio-PVC film as prepared in Example IV	9.61	12.9	35.2
Negative Control (PE) (25 grams)	21.4	28.6	78.5
Positive Control (Cellulose) (20 grams)	8.8	11.8	32.3

Gas Production Data - Samples

Day	Bio-PVC film as prepared in Example IV			Negative Control PE			Positive Control Cellulose			Inoculum Control		
	A	B	C	A	B	C	A	B	C	A	B	C
Totals 1-30	5819	5683	4572	1710	971	1019	17457	18409	16671	1686	1550	1650
31	75	83	73	9	22	24	66	97	107	9	12	12
32	75	83	73	9	22	24	66	97	107	9	12	12
33	75	83	73	9	22	24	66	97	107	9	12	12
34	75	50	95	25	30	25	325	250	245	60	50	50
35	75	50	95	25	30	25	325	250	245	60	50	50
36	89	71	84	9	19	7	173	138	148	37	24	17
37	89	71	84	9	19	7	173	138	148	37	24	17
38	89	71	84	9	19	7	173	138	148	37	24	17
39	89	71	84	9	19	7	173	138	148	37	24	17
40	91	98	98	7	35	28	278	165	116	35	28	70
41	91	98	98	7	35	28	278	165	116	35	28	70
42	91	98	98	7	35	28	278	165	116	35	28	70
43	21	39	35	19	11	21	55	39	49	23	19	9
44	21	39	35	19	11	21	55	39	49	23	19	9
45	21	39	35	19	11	21	55	39	49	23	19	9
Totals 31-45	1067	1044	1144	191	340	297	2539	1955	1898	469	373	441
Avg 31-45	1085			276			2131			428		

Methane and Carbon Dioxide Readings

Day	Bio-PVC film as prepared in Example IV		Negative Control PE		Positive Control Cellulose		Inoculum Control	
	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide
35	20 %	19.3 %	9 %	8.1 %	21 %	17.6 %	7 %	4.8 %
40	21 %	17.4 %	7 %	5.4 %	25 %	14.5 %	5 %	4.5 %
45	17 %	15.6 %	4 %	3.9 %	12 %	9.1 %	5 %	4.5 %
Avg	19.3 %	17.4 %	6.7 %	5.8 %	19.3 %	13.7 %	5.7 %	4.6 %

Calculations of Results

Sample	Average Weight grams	Average Gas Vol. (mL)	Methane			Carbon Dioxide			Total CH ₄ +CO ₂	Sample-Inoculum=
			(%)	(mL)	(Wt) C (grams)	(%)	(mL)	(Wt) C (grams)		
Bio-PVC film as prepared in Example IV	25	1085	19.3	209	0.11	17.4	189	0.1	0.21	0.19
Negative Control PE	25	276	6.7	19	0.01	5.8	16	0.009	0.019	0
Positive Control	20	2131	19.3	411	0.22	13.7	292	0.16	0.38	0.36
Inoculum Control	1000	428	5.7	24	0.01	4.6	20	0.01	0.02	

Results (Average of 3)				
	Gaseous Carbon Recovered	Theoretical Grams	(%) Biodegradation Days 31-45	(%) Biodegradation Days 1-45
Bio-PVC film as prepared in Example IV	0.19	9.61	1.98 %	9.57 %
Negative Control PE	0	21.4	0 %	0 %
Positive Control	0.36	8.8	4.09 %	87.39 %

The results support the suitability of the product for the blister packing application. It also depicts the biodegradation of the film prepared in accordance with the process of the present disclosure.

Example V:

A. Batch Mixing Operation:

407.00 kg of PVC homopolymer suspension resin, 24.700 kg of Vinyl chloride/vinyl Acetate Copolymer, 6.900 kg of Polymer stabilizer, 37.00 kg of Acrylic polymer impact modifier, 1.480 kg of Montanic ester wax, 6.160 kg of epoxidized soyabean oil, 6.160 kg of Partial esters of fatty acids with glycerol, 2.460 kg of butadiene/ methylmethacrylate/ styrene processing aid, 1.230 kg of Bis-stearoyl-ethylenediamine, 1.230 kg of PolyVinyl chloride, 1.230 kg of Acrylic polymer processing aid, 0.988 kg of Polyadipate, 0.367 kg of Mg-Silicate based talc and 3.480 kg of Bio Pro-degradant (Eco-pureTM manufactured by Bio-tech Environmental, LLC) were added to the batch mixing system from the respective storage system using programmable logic controlled material dosage/discharge systems and mixed to obtain a thoroughly mixed batch of ingredients.

B. Calender Operation:

The following process protocol was followed for carrying out the calendering operation.

PROCESS STEP	EQUIPMENT	PARAMETER		UNIT	VALUE
Extruder	Ko-Kneader	Difference between the torque of the feeder screw and the torque of the output screw		%	11.54
		Screw speed		rpm	8.5
Calendering		Temperature	Cylinder 1	°C	175
		Speed		m/min	13.7
		Temperature	Cylinder 2	°C	177
		Speed		m/min	15.1
		Temperature	Cylinder 3	°C	190
		Speed		m/min	16.6
		Temperature	Cylinder 4	°C	205
		Speed		m/min	19.5
		Temperature	Cylinder 5	°C	175
		Speed		m/min	24.8
Winder		Web tension		(dN)	250
Stenter		Temperature	Heat Zone 1	°C	115
			Heat Zone 2	°C	102
			Heat Zone 3	°C	100
			Stretch Zone1	°C	99
			Stretch Zone 2	°C	98

The same manufacturing process as in the example 1 is followed till the calendering stage. Further the film is heated and stretched in both directions to make it a thinner film. These films are used for shrink packaging applications.

C. Test Data

The film thus produced is tested for its biodegradability

Results:

Determining anaerobic biodegradation of plastic materials under high solids anaerobic digestion conditions

Inoculum Source:

- Organic Compost – McEnroe Organic Farms, Millerton, NY
Mattabassit Waste Treatment Facility Anaerobic

Digestion

❖ Solid Content	22%
❖ pH	8.2
❖ Volatile Fatty Acids	0.7 g/kg
❖ Ammonia Nitrogen	1.0 mg/kg
❖ Volatile Solids	24.9 %

Procedure: The procedure followed was the same as described in Example I

The results are shown in the following tables.

Theoretical Gas Production

Samples	Carbon Content (grams)	Methane (grams)	Carbon Dioxide (grams)
Bio-PVC film as prepared in Example V	9.61	12.9	35.2
Negative Control (PE) (25 grams)	21.4	28.6	78.5
Positive Control (Cellulose) (20 grams)	8.8	11.8	32.3

Gas Production Data - Samples

Day	Bio-PVC film as prepared in Example V			Negative Control PE			Positive Control Cellulose			Inoculum Control		
	A	B	C	A	B	C	A	B	C	A	B	C
Totals 1-30	5267	4823	4635	1525	2063	1664	17525	18001	15342	3140	2805	2711
Totals 31-45	901	1100	852	247	220	215	1198	1377	1441	300	266	319
Avg 31-45	951			227			1339			295		

Methane and Carbon Dioxide Readings

Day	Bio-PVC film as prepared in Example V		Negative Control PE		Positive Control Cellulose		Inoculum Control	
%	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide	Methane	Carbon Dioxide
34	18 %	19.9%	5%	3.8 %	29 %	26.8 %	7 %	5.4 %
38	16 %	15.9 %	3 %	3.5 %	30 %	29.2 %	3 %	2.9 %
45	16 %	15.9 %	3 %	3.5 %	27 %	24.3%	4 %	3.4 %
Avg	16.7 %	17 %	3.7 %	3.5 %	28.7 %	26.8 %	4.7%	3.9 %

Calculations of Results

Sample	Average Weight grams	Average Gas Vol. (mL)	Methane			Carbon Dioxide			Total CH ₄ + CO ₂	Sample-Inoculum=
			(%)	(mL)	(Wt) C (grams)	(%)	(mL)	(Wt) C (grams)		
Bio-PVC film as prepared in Example V	25	951	16.7	159	0.09	17	162	0.09	0.18	0.17

Negative Control PE	25	227	3.7	8	0.004	3.5	8	0.004	0.008	0
Positive Control	20	1339	28.7	384	0.21	26.8	359	0.19	0.40	0.39
Inoculum Control	1000	295	4.7	14	0.008	3.9	12	0.006	0.004	

Results (Average of 3)				
	Gaseous Carbon Recovered	Theoretical Grams	(%) Biodegradation Days 31-45	(%) Biodegradation Days 1-45
Bio-PVC film as prepared in Example V	0.17	9.61	1.77%	10.1%
Negative Control PE	0	21.4	0 %	0 %
Positive Control	0.39	8.8	4.43 %	84.65 %

The results support the suitability of the product for the blister packing application. It also depicts the biodegradation of the film prepared in accordance with the process of the present disclosure.

Example VI

The film produced as per the example I is kept at temperature and Humidity conditions of 40 °C and 75% RH for testing the possibility of any microbial growth or yield or mould growth due to the biodegradability characteristics of the film for testing its applicability in food and pharma contact materials.

Testing procedure

1. Sampling Locations

The films produced as per example 1 were kept at environmental chamber maintaining 40 °C and 75% RH . Films were periodically withdrawn from the chambers and tested for any microbial, mould or yeast growth. In Parallel, a non bio-degradable sample was also studied under the same condition as the reference sample.

2. Sampling & Analysis methodology

a) Total plate count and Total yeast & mould count (Swab Sample)

Sampling Technique

A Sterile Swab was being moistened in the swab head. Rub the swab head slowly and thoroughly over approximately 100cm² (with a 10x10cm sterile template) of surface three times, reversing direction between strokes.

Pour Plate Technique

For total bacterial count: Pipette 1ml of liquid from the phosphate buffer solution in to a sterile petri dish. Add sterile Trypticase Soy Agar (TSA) into the inoculated dish, rotate and allowed to solidify and was then incubated or 48 hours at 35°C. For Total yeast &

mould count add sterile Potato Dextrose Agar (PDA) into the inoculated dish and was then incubated for 120 hours at 25 °C.

Results are tabulated in Table 1.

Table 1: Comparative microbial test results of 250 Micron Bio -PVC White Opaque film prepared in example 1 and 300 Micron PVC Glass Clear, Bilcare

Sr No.	Product	Number of Months (Mths)	Total Bacterial Counts (CFC/100cm ²)	Total Yeast & Mould Counts (CFC/100cm ²)
1	250 Mics bio-PVC White Opaque as prepared in example 1	1	<10	<10
2	300 Mics PVC Glass Clear	1	<10	<10
3	250 Mics bio-PVC White Opaque as prepared in example 1	2	<10	<10
4	300 Mics PVC Glass Clear	2	<10	<10
5	250 Mics bio-PVC White Opaque as prepared in example 1	3	<10	<10

6	300 Mics PVC Glass Clear	3	<10	<10
7	250 Mics bio- PVC White Opaque as prepared in example 1	4	<10	<10
8	300 Mics PVC Glass Clear	4	<10	<10
9	250 Mics bio- PVC White Opaque as prepared in example 1	5	<10	<10
10	300 Mics PVC Glass Clear	5	<10	<10
11	250 Mics bio- PVC White Opaque as prepared in example 1	6	<10	<10
12	300 Mics PVC Glass Clear	6	<10	<10

Form the test results it is seen that the Bacterial and Yeast & Mould counts were not detected in both the biodegradable film as well as the reference film. This proves that the invented film is biodegradable only at land filling, anaerobic conditions and no microbial growth is possible at the regular storage condition. This makes the film suitable for the food and drug contact application though it is capable of biodegradation after use.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “a”, “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

The numerical values given for various physical parameters, dimensions and quantities are only approximate values and it is envisaged that the values higher or lower than the numerical value assigned to the physical parameters, dimensions and quantities fall within the scope of the disclosure and the claims unless there is a statement in the specification to the contrary.

While certain embodiments of the inventions have been described, these embodiments have been presented by way of examples only, and are not intended to limit the scope of the disclosure. Variations or modifications in the combination of this invention, within the scope of the disclosure, may occur to those skilled in the art upon reviewing the disclosure herein. Such variations or modifications are well within the spirit of this disclosure. The accompanying claims and their equivalents are intended to cover such forms or modifications as would fall within the scope and spirit of the disclosure.

Claims:

1. A process for preparing a bio-degradable PVC based pharmaceutical grade thermo-formable film, said process comprising the following steps:
 - a. mixing pharmaceutical grade PVC resin, copolymer, at least one impact modifier, bio pro-degradant, at least one processing aid, and at least one stabilizer in a mixer to obtain a mixed batch of ingredients;
 - b. extruding the mixed batch of ingredients in an extruder at a screw speed ranging between 2 rpm and 15 rpm and temperature ranging between 55 °C and 70 °C to obtain fluxed polymeric flakes; and
 - c. calendering the polymeric flakes by subjecting them to at least two calender rolls maintained at temperatures ranging between 100 °C and 250 °C to obtain a bio-degradable PVC based pharmaceutical grade thermo-formable film,wherein, said film is stable in aerobic conditions and is bio-degradable under anaerobic conditions.
2. The process as claimed in claim 1, wherein the method step of mixing further comprises adding at least one pigment in the mixed batch.
3. The process as claimed in claim 1, wherein the method step of mixing further comprises adding titanium dioxide in the mixed batch.
4. The process as claimed in claim 1, wherein the method step of extruding comprises maintaining a predetermined percentage

difference between the torque of the feeder screw and torque of the output screw of the extruder which causes generation of pressure and heat on the mixture, resulting in fluxing of the material.

5. The process as claimed in claim 4, wherein the percentage difference between the torque of the feeder screw and torque of the output screw ranges between 5 and 20, preferably, 8 and 16.
6. The process as claimed in claim 1, wherein the pharmaceutical grade PVC resin is at least one selected from the group consisting of PVC suspension resin and PVC homopolymer suspension resin.
7. The process as claimed in claim 1, wherein the copolymer is Vinyl Chloride/Vinyl Acetate copolymer.
8. The process as claimed in claim 1, wherein the impact modifier is at least one selected from the group consisting of methylmethacrylate-butadiene-styrene-acrylic copolymer and acrylic modifier.
9. The process as claimed in claim 1, wherein the bio pro-degradant is Ethylene-Vinyl Acetate copolymer with organoleptic additives.
10. The process as claimed in claim 1, wherein the amount of bio pro-degradant ranges between 0.01 % and 20 % with respect to the mass of the film, preferably, 0.1 % and 10.0 % with respect to the mass of the film.

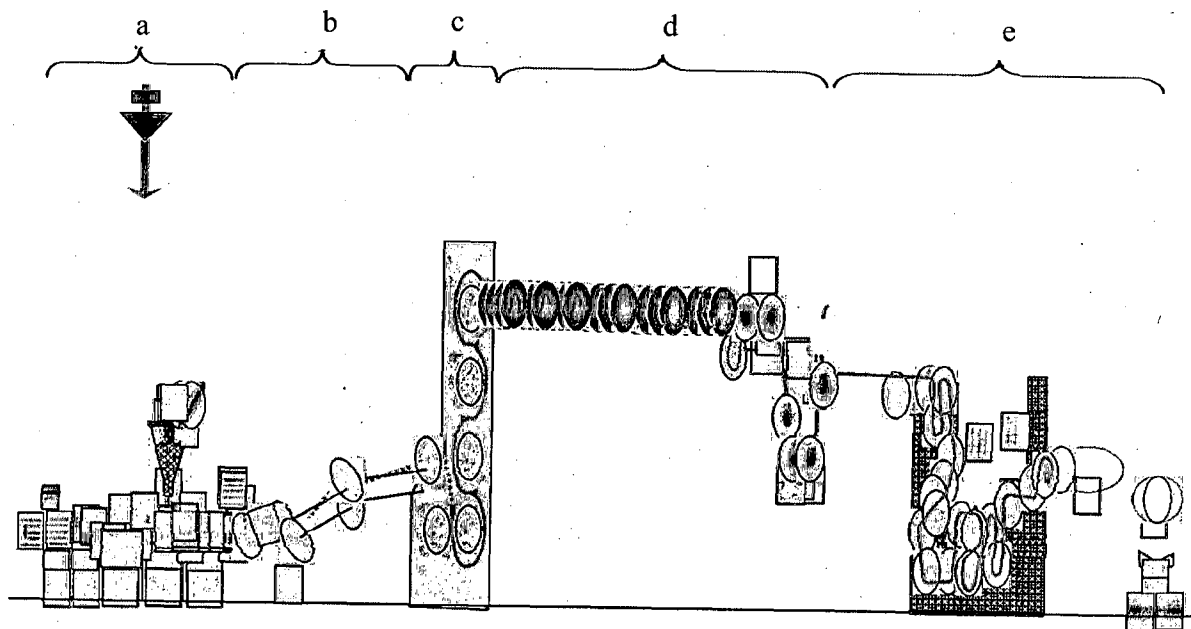
11. The process as claimed in claim 1, wherein the processing aid is at least one selected from the group consisting of anti-blocking/slipping agents, antistatic agents, lubricants, release agents, anti-sticking agents and melt strength/viscosity balancing agents.
12. The process as claimed in claim 1, wherein the stabilizer is at least one selected from the group consisting of polymer and soyabean stabilizer.
13. The process as claimed in claim 1, wherein the two calender rolls are arranged at a distance ranging between 0.01 mm and 50 mm from each other.
14. The process as claimed in claim 1, wherein the calender rolls are arranged in a cross-axial or bending position.
15. A bio-degradable PVC based pharmaceutical grade thermo-formable film obtained by a process of claim 1, said film comprising:
 - i. a pharmaceutically grade PVC resin, ii. a copolymer, iii. at least one impact modifier, iv. A bio pro-degradant, v. at least one processing aid, vi. optionally, a titanium dioxide, vii. at least one stabilizer and viii optionally, at least one pigment,wherein, said film is stable in aerobic conditions and is bio-degradable under anaerobic conditions.

16. The film as claimed in claim 15, wherein the pharmaceutical grade PVC resin is at least one selected from the group consisting of PVC suspension resin and PVC homopolymer suspension resin.
17. The film as claimed in claim 15, wherein the copolymer is Vinyl Chloride/Vinyl Acetate copolymer.
18. The film as claimed in claim 15, wherein the impact modifier is at least one selected from the group consisting of methylmethacrylate-butadiene-styrene-acrylic copolymer and acrylic modifier.
19. The film as claimed in claim 15, wherein the bio pro-degradable is Ethylene-Vinyl Acetate copolymer with organoleptic additives.
20. The film as claimed in claim 15, wherein the amount of bio pro-degradable ranges between 0.01 % and 20 % with respect to the mass of the film, preferably, 0.1 % and 10.0 % with respect to the mass of the film.
21. The film as claimed in claim 15, wherein the processing aid is at least one selected from the group consisting of anti-blocking/slipping agents, antistatic agents, lubricants, release agents, anti-sticking agents and melt strength/viscosity balancing agents.
22. The film as claimed in claim 15, wherein the stabilizer is at least one selected from the group consisting of polymer and soyabean stabilizer.

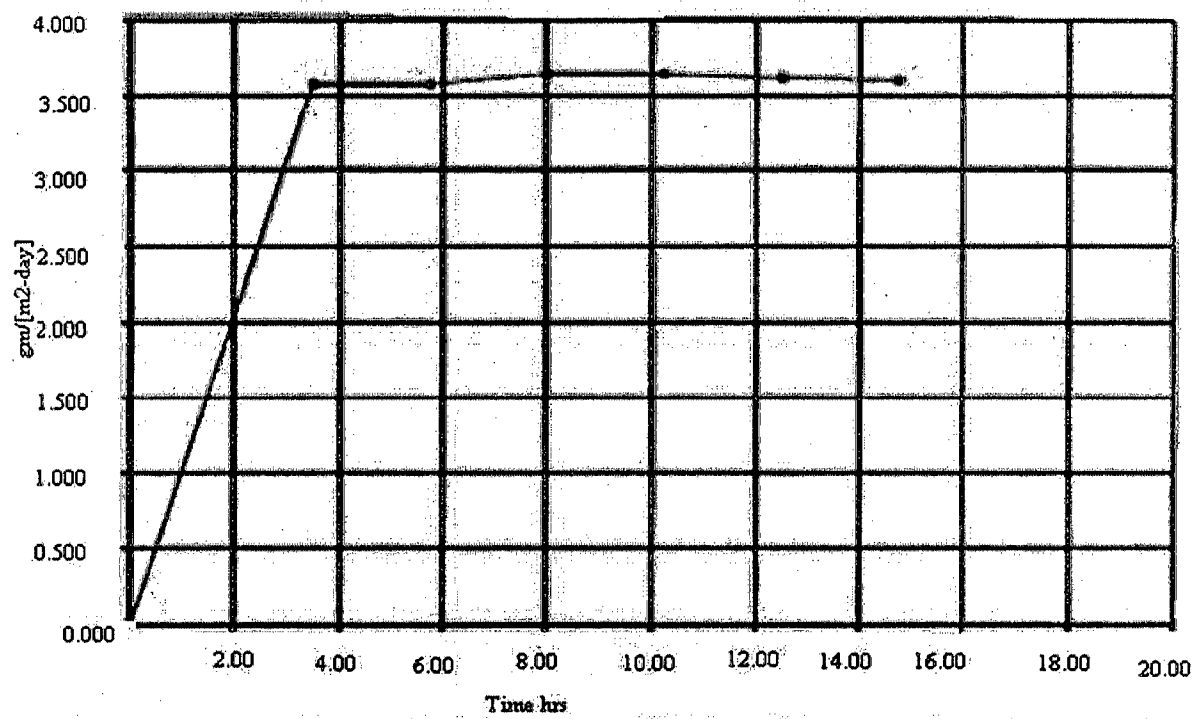
23. The film as claimed in claim 15, wherein the PVC film is rigid.

24. A blister container for packing pharmaceutical products made using the film in accordance with claim 15.

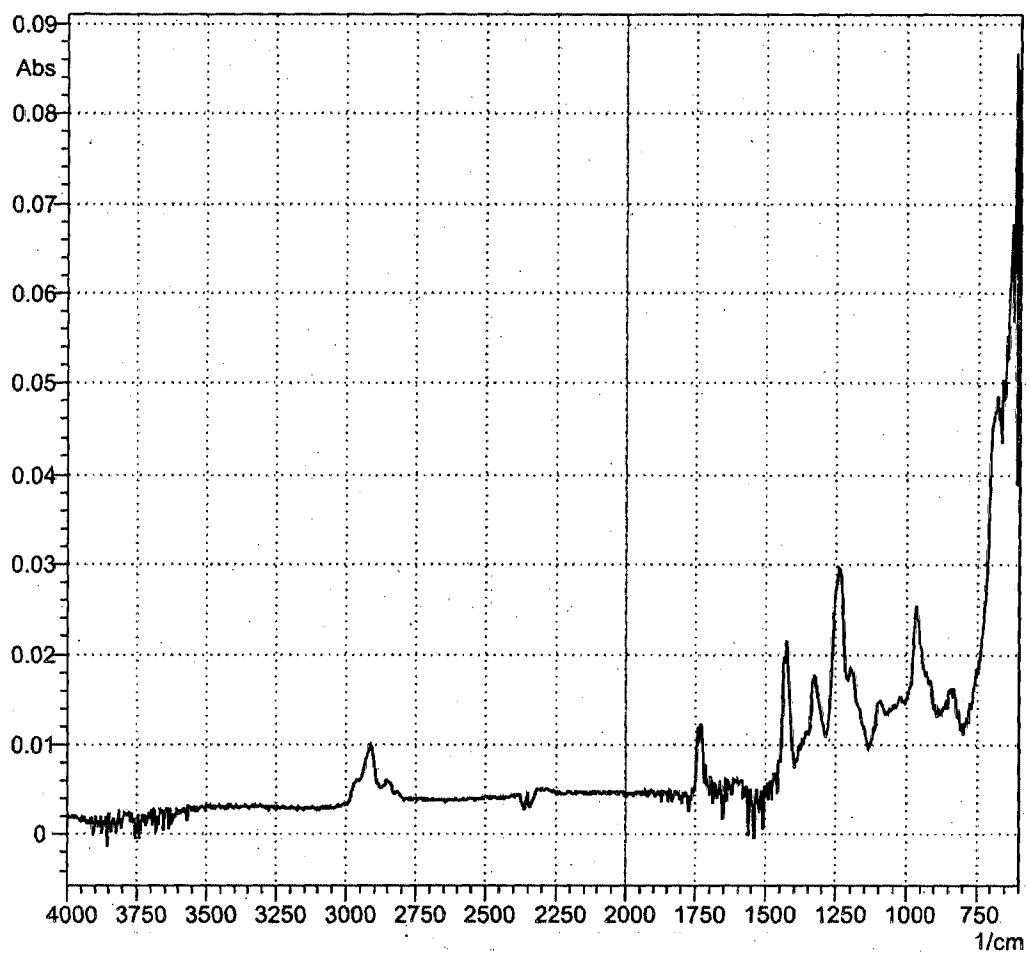
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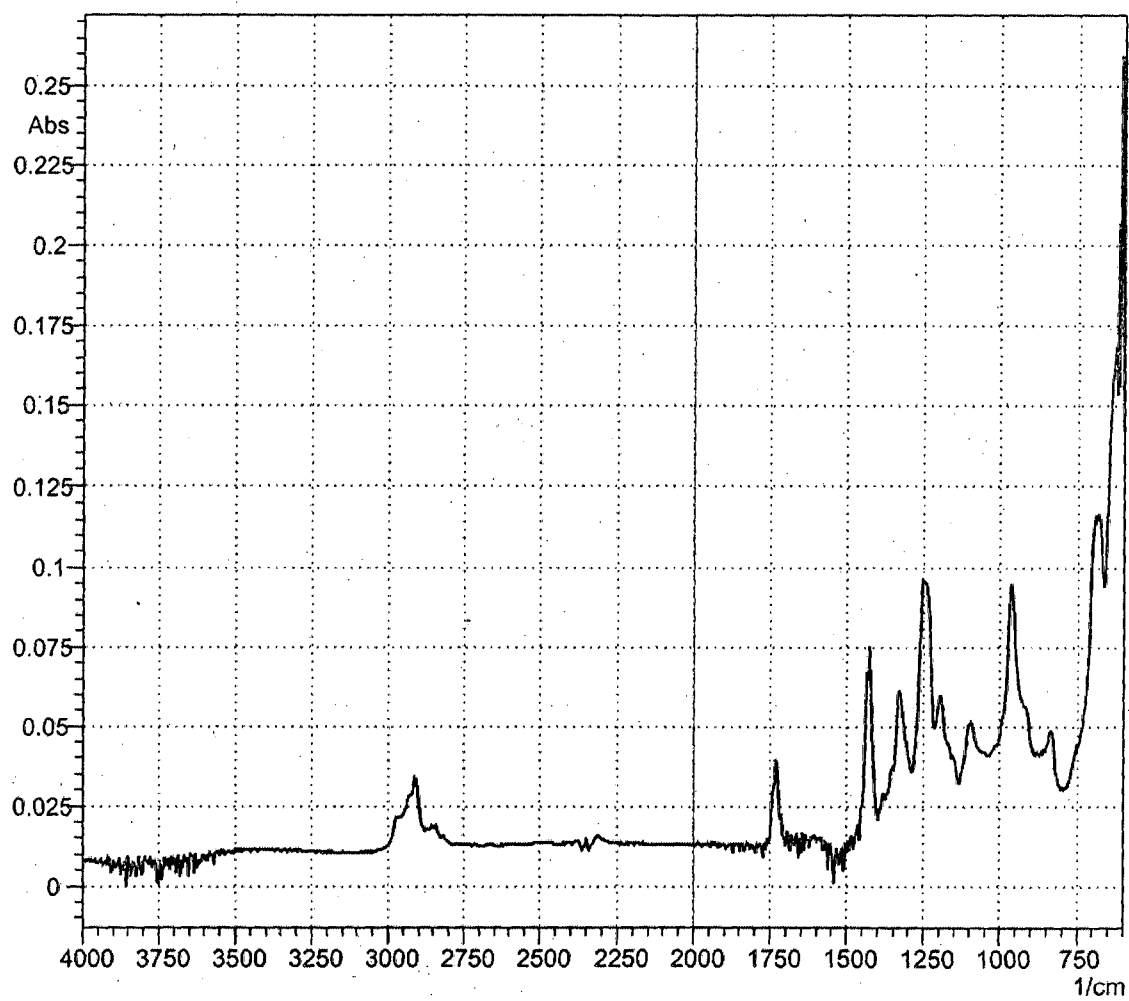
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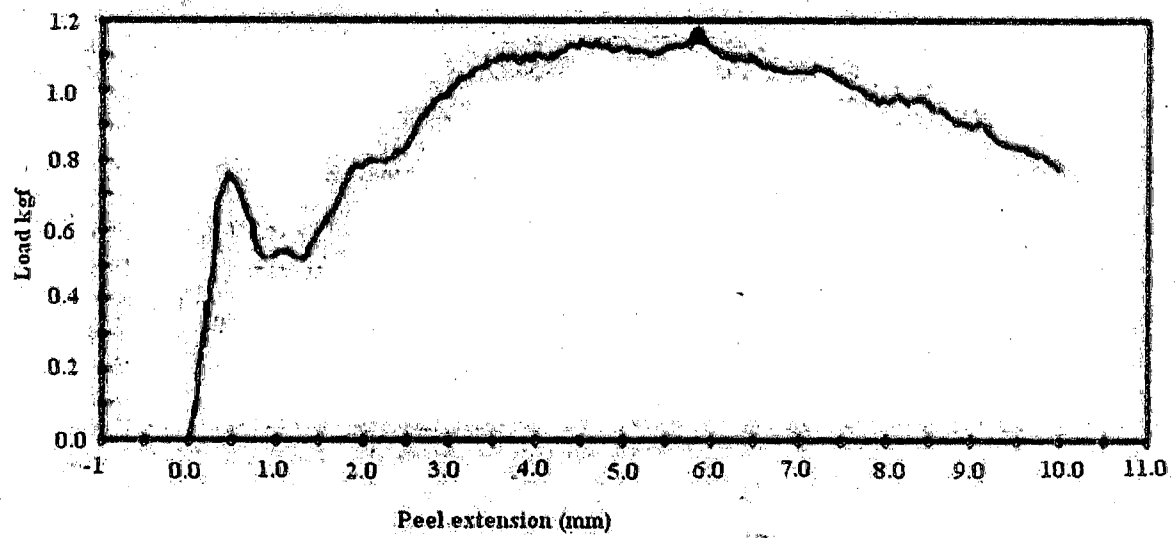
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