

Brigard & Castro
Abogados

Apartado 3692

Cra 7 16 -56
Bogotá - Colombia

AS 101 100

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
Rad 06-042613- -00001-0000
Fec 2008-07-28 16:13:32 Dep 2020 NUECREACIONES
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Señores

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

DIVISION DE NUEVAS CREACIONES

E S D

RESPUESTA A OFICIO 6399 DE JUNIO 08, 2008

REF Expediente No 06 042 613 - Solicitud de Patente

a favor de **MICHIGAN STATE UNIVERSITY**

Titulada **COPOLÍMEROS DE INJERTO DE ACEITE VEGETAL-ALMIDÓN Y SUS
COMPUESTOS DE BIOFIBRA, Y UN PROCESO PARA SU MANUFACTURA**

JUAN PABLO CADENA SARMIENTO, vecino de Bogotá, portador de la tarjeta profesional No 57492, obrando como apoderado sustituto de la sociedad **MICHIGAN STATE UNIVERSITY**, según lo acredita el documento de sustitución de poder anexo, para dar cumplimiento al requerimiento notificado por fijación en lista el 08 de Junio de 2006, de acuerdo con los Artículos 38 y 39 de la Decisión 486 de la Comisión de la Comunidad Andina, me permito allegar

- 1 Fotocopia autenticada del documento de poder confendo por **MICHIGAN STATE UNIVERSITY**, incluyendo el Certificado que acredita la Existencia y Representación Legal, debidamente apostillado, junto con su correspondiente traducción
- 2 Documento donde consta la cesión del derecho a la Patente otorgado por los inventores al solicitante, debidamente apostillado, junto con su correspondiente traducción
- 3 Copia certificada por la Oficina de Patentes de Estados Unidos de la primera solicitud presentada para la misma invención, de la cual **REIVINDICAMOS PRIORIDAD**, junto con su correspondiente traducción al castellano
- 4 Extracto para publicación y Tarjeta de archivo Temático

De la señora Jefe atentamente,


JUAN PABLO CADENA SARMIENTO
C C No 80 410 337 de Usaquén
Cra 7 No 16-56 Piso 9

Bogotá Julio 27 2008
CHP/MEV/her

Extracto de P 102



Industria y Comercio
SUPERINTENDENCIA

SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISION DE NUEVAS CREACIONES
TARJETA ARCHIVO TEMÁTICO

PATENTE DE INVENCION ☒ PCT ☐

MODELO DE UTILIDAD ☐

DISEÑO INDUSTRIAL ☐

(21) N° de solicitud 08 042 813

(541) Clasificación Internacional

(71) Solicitante(s) MICHIGAN STATE UNIVERSITY

(72) Inventor(es) Ramani Narayan Sunder Balakrishnan, Boo-Young Shin

(74) Agente JUAN PABLO CADENA SARMIENTO

(30) Prioridad

(31) No Prioridad 11/124,491

(32) Fecha 08-05-2005

(33) País US

(85) Fecha Inicio fase nacional

(86) Datos relativos a la presentación de la solíc. PCT
Fecha de presentación de la solicitud
No. de solicitud PCT/

(87) Publicación Internacional
Fecha
No Publicación WO

FIGURA CARACTERISTICA
6 x 6 cm

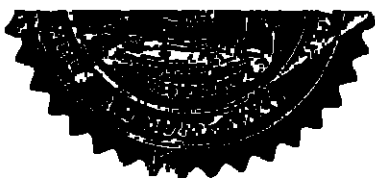
(54) Título COPOLIMEROS DE INJERTO DE ACEITE VEGETAL-ALMIDÓN Y SUS COMPUESTOS DE BIOFIBRA, Y UN PROCESO PARA SU MANUFACTURA

Reivindicación(es) 1 Una composición de copolímero de Injerto de aceite vegetal - almidón, biodegradable, que es una mezcla reactivamente combinada, que comprende un almidón granulado, un aceite vegetal o su derivado, que comprende un modificador reactivo con el almidón, y un plastificante hidroxilado

CONTINUA AL RESPALDO

100

- 10 La composición de la Reivindicación 1, en donde el plastificante es un alcohol polihídrico en una cantidad entre 10 a 40% en peso del peso total de la composición
- 18 La composición de la Reivindicación 1, que comprende el modificador el cual se selecciona del grupo conformado por ácido dibásico o cíclico y sus anhídridos
- 22 Un proceso para producir una composición de copolímero de aceite vegetal – almidón, biodegradable, que comprende
 - (a) Combinar reactivamente una mezcla de un almidón granulado, un aceite vegetal o su derivado, que comprende un modificador reactivo con el almidón, y un plastificante en un reactor; y
 - (b) Retirar la composición del reactor para producir la composición



Brigard & Castro
BOGOTA COLOMBIA

PODER

POWER OF ATTORNEY

AS 101100
101

Yo (nosotros) MICHIGAN STATE UNIVERSITY

I (We) MICHIGAN STATE UNIVERSITY

domiciliado(s) en 238 Administration Building, East Lansing, Michigan 48824, Estados Unidos de América

domiciled in 238 Administration Building, East Lansing, Michigan 48824 United States of America

Nombro(amos) a JUAN PABLO CADENA SARMIENTO y/o CARLOS H PALACIO EASTMAN y/o MARTIN E TORRES CARDOZO de Bogotá, Colombia, apoderados verdaderos y legales con poder especial, amplio y suficiente, para recabar de las oficinas y autoridades colombianas administrativas, judiciales y de policía en cualquier instancia o recurso, la obtención de patentes de invención, registros de marcas, diseños industriales, modelos de utilidad, depósitos de nombres comerciales, lemas comerciales y enseñas, registros de derechos de autor y contratos sobre los mismos, obtención de certificados de obtentor de variedades vegetales, así como sus prórrogas renovaciones anotaciones de traspaso, cambios de nombre y/o domicilio elaborar tramitar y registrar contratos de licencia y licencias de cualesquiera clases, intervenir como demandante o como demandado en cualquier instancia y recurso ante cualquier juez corporación, funcionario público o autoridad en acciones judiciales administrativas y de policía tales como oposiciones al registro de marcas acciones de caducidad, cancelación y nulidad acciones reivindicatorias, acciones derivadas de la obtención o explotación de licencias, acciones de competencia desleal, demandas penales, acciones de indemnización de perjuicios reclamos y observaciones a solicitudes de patentes diseños industriales modelos de utilidad, en el ejercicio de medidas cautelares y en otras acciones o medidas similares

Hereby appoints JUAN PABLO CADENA SARMIENTO and/or CARLOS H PALACIO EASTMAN and/or MARTIN E TORRES CARDOZO, of Bogotá, Colombia, my (our) true and lawful attorneys with special, ample and sufficient power to obtain from the Colombian administrative, judicial and police offices and authorities, in any instance, privileges of patents of invention, registration of trademarks, industrial designs and utility models registration of commercial names, slogans and emblems, registration of copyright and contracts related thereto, certificates of obtainers of vegetables varieties, as well as extensions, renewals and recordal of assignments and changes of name and domicile related to the above, to prepare, prosecute and register licenses of any kind, to act as plaintiff or defendant in any instance or recourse, and before any judge, corporation public official or authority in judicial, administrative and police actions such as oppositions to the registration of trademarks, caducity, cancellation and nullity actions, claim actions derived from the obtaining or exploitation of licenses action of unfair competition, criminal proceedings, actions for payment of damages claims or observations on patent applications, models, industrial designs and utility models, in the exercise of preventive measures and in other actions or similar proceedings

A este efecto, lo(s) faculto(amos) para elevar solicitudes formular descripciones enmiendas, protestas, declaraciones, oposiciones cancelaciones nulidades, apelaciones y reclamos, pagar impuestos, cuotas y gravámenes prescritos por la ley, renunciar o desistir a derechos concedidos o en curso de concesión, recibir toda clase de documentos y valores, dando el descargo respectivo, y, en general, para dar ante dichas autoridades todos los pasos necesarios al efecto indicado y tomar todas las medidas que creyeran conducentes al resguardo de mis (nuestros intereses), con todas las demás facultades legales necesarias

To that effect they hereby empowered to file applications, formulate descriptions amendments protests declarations, oppositions, cancellations, nullities, appeals and claims, to pay taxes, quotas and assessments prescribed by law renounce or waive the rights granted or in the process of granting, to receive all kinds of documents and valuables, giving the respective release of receipt, and, in general, to take before said authorities the necessary steps to such effect, and any measures that they may deem pertinent to the safeguard of my (our) interest(s) with all the other necessary legal powers

Este poder comprende la facultad de ratificar o confirmar en mi (nuestro) nombre lo actuado así como la facultad de desistir, transigir, comprometer, y la de sustituirlo en todo o en parte y revocar sustituciones Asimismo nombro(amos) a JUAN PABLO CADENA SARMIENTO y/o CARLOS H PALACIO EASTMAN y/o MARTIN E TORRES CARDOZO domiciliado(s) en la Carrera 7 No 16 56 Piso 9 mls (nuestros) apoderados en Colombia, con facultades para recibir notificaciones y designar apoderados judiciales o extrajudiciales

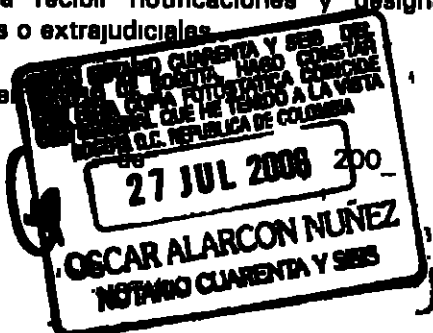
This power includes the faculty to ratify or confirm desist settle and compromise on my (our) behalf, and to substitute this power in whole or in part and to revoke substitutions At the same time I (We) hereby appoint JUAN PABLO CADENA SARMIENTO and/or CARLOS H PALACIO EASTMAN and/or MARTIN E TORRES CARDOZO domiciled in Carrera 7 No 16-56, Piso 9, my (our) attorney(s) in fact, with powers to receive notifications and to designate judicial and extrajudicial attorneys

Otorgado y firmado en

hoy de

Firma

Nombre
Cargo



Granted and signed in East Lansing, Michigan

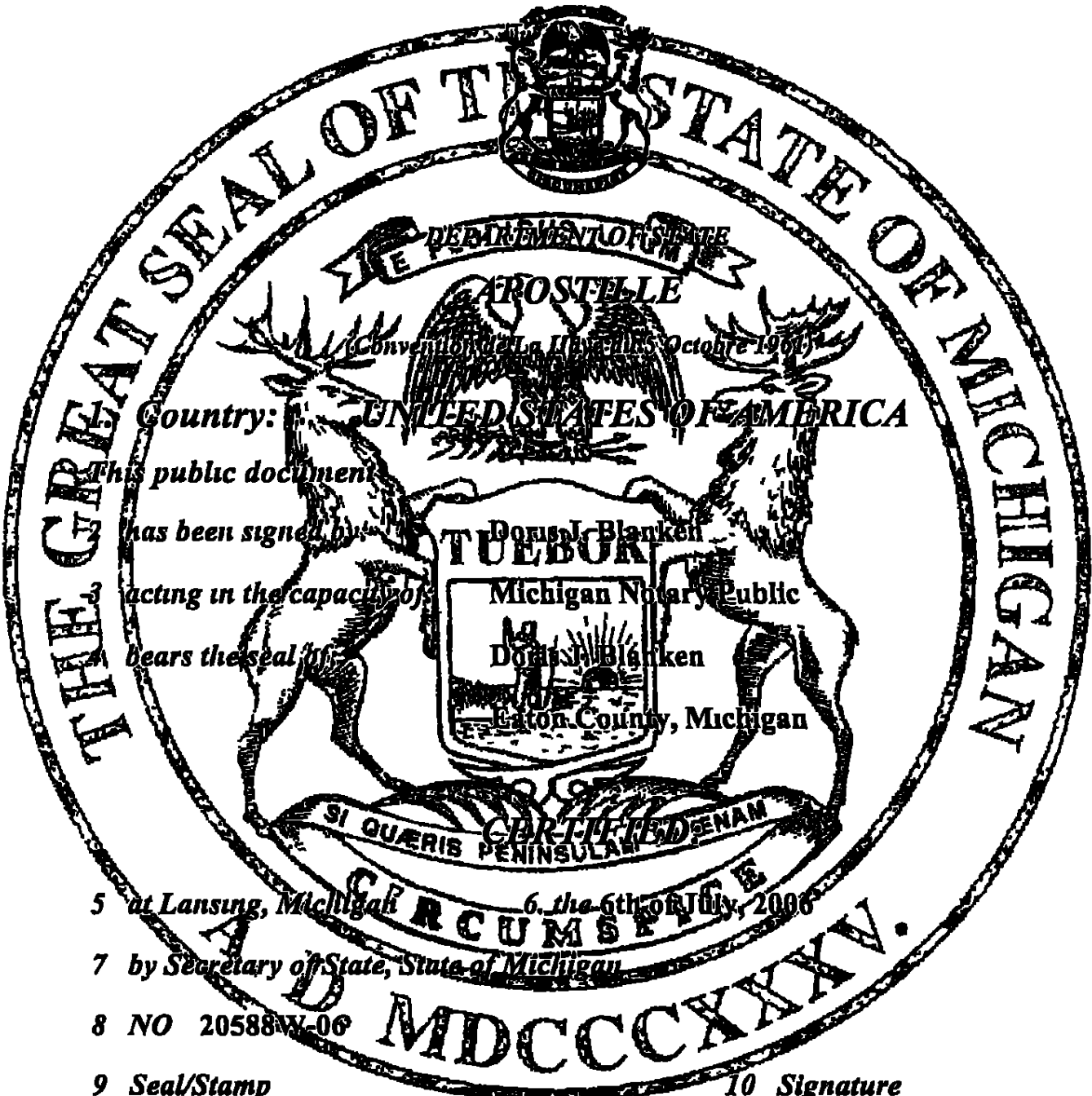
this 21st day of June 2006

Signature, *Loraine J Hudson*

Name Loraine J Hudson
Title Director of Intellectual Property

State of Michigan

102



Terri Lynn Land

Terri Lynn Land

This certification attests only to the authenticity of the signature of the official who signed the official document, the capacity in which that official acted, and where appropriate, the identity of the seal or stamp which the document bears. This certification is not intended to certify that the contents of the document are correct, nor that the document is a true and correct copy of the original of the State of Michigan.



CERTIFICADO NOTARIAL (SOCIEDAD)

NOTARIAL CERTIFICATE (CORPORATION)

Hoy de
personalmente compareció ante mí

a quien conozco, y de quien me consta que firmó el anterior documento debidamente juramentado declaró y dijo que él (ella) es

de MICHIGAN STATE UNIVERSITY

Sociedad legalmente constituida para un período de duración que aun no ha terminado, que el declarante está autorizado por dicha compañía para conferir este poder, que el sello estampado por él al pie de tal poder, en nombre y representación de la expresada compañía, es el sello oficial de ella, y que el objeto para el cual se confiere este instrumento está comprendido dentro del objeto social de la sociedad

El suscrito Notario hace constar que tuvo a la vista las pruebas de la existencia de la sociedad

Estado de
con fecha

y que quien confiere el presente poder es su representante

Notario Público (sello)

**CERTIFICADO NOTARIAL
PERSONA NATURAL**

Hoy de de , compareció(eron)
ante mí

a quien (es) conozco y me consta que es(son) la(s) persona(s) firmante(s) del documento que precede y quien(es) declara(n) que otorgan el mismo para los fines y propósitos que en él se expresan

Notario Publico (seal)

On this 21 day of June, 2006 of
personally appeared before me

LORAIN J HUDSON

to me known and known to me to be the person who signed the foregoing instrument who being by me duly sworn did depose and say that he (she) is the DIRECTOR OF INTELLECTUAL PROPERTY

of MICHIGAN STATE UNIVERSITY

Which is legally constituted for a term not yet expired, that deponent is authorized by said Company to confer this Power of Attorney that the seal affixed hereunto by the deponent in the name and on behalf of said Corporation is the corporate seal thereof and that the purpose for which this instrument is granted is included within the corporate purpose of company

The undersigned Notary Public attests that he has seen the documents that prove the existence of the company

State of
under date of

and that he (she) who grants the present power of attorney is its Representative

Public Notary (Seal)

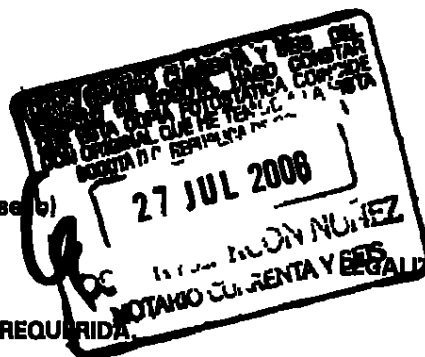
**NOTARIAL CERTIFICATE
NATURAL PERSON**

On this 21st day of June of 2006
personally appeared before me

who is(are) known to me and who I attest executed the foregoing document and he (they) declared he (they) grant(s) the document for the uses and purposes therein expressed

Notary Public (Seal)

Doris J. Blanken



LEGALIZACION POR APOSTILLA ES REQUERIDA.

DORIS J BLANKEN
Notary Public, Eaton County, MI
My Comm Expires June 13, 2007

X 101

**TRADUCCION AL ESPAÑOL del Documento de Poder otorgado por
MICHIGAN STATE UNIVERSITY domiciliada en 238 Administration
Building, East Lansing, Michigan 48824, Estados Unidos de América**

ESTADO DE MICHIGAN

DEPARTAMENTO DE ESTADO

APOSTILLA

(Convención de La Haya del 5 de octubre de 1961)

1 País ESTADOS UNIDOS DE AMÉRICA

Este documento publico

2 ha sido firmado por Doris J Blanken

3 actuando en capacidad de Notario público de Michigan

**4 lleva el sello de Doris J Blanken
Condado de Eaton, Michigan**

CERTIFICADO

5 en Lansing, Michigan 6 el 6 de julio de 2006

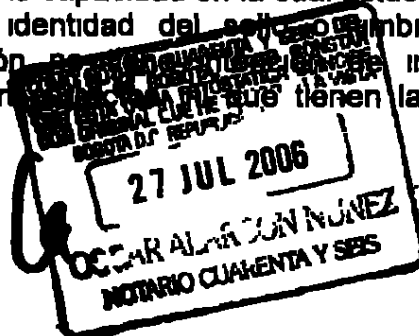
7 por el Secretario de Estado, Estado de Michigan

8 NO 20588W-06

9 Sello/timbre 10 Firma

**(Firma)
Terri Lynn Land**

La certificación da testimonio de la autenticidad de la firma del funcionario que firmó el documento anexo, la capacidad en la cual actuó ese funcionario, y donde sea apropiado, la identidad del sello o timbre que lleva el documento. Esta certificación no implica que los contenidos del documento son correctos o que tienen la aprobación del Estado de Michigan.



State of Michigan



DEPARTMENT OF STATE

APOSTILLE

(Convention de La Haye du 5 Octobre 1961)

1. Country: **UNITED STATES OF AMERICA**

This public document

2. has been signed by: **Tammi L. Taylor**

3 acting in the capacity of: **Michigan Notary Public**

4. bears the seal of: **Tammi L. Taylor**
Ingham County, Michigan

CERTIFIED:

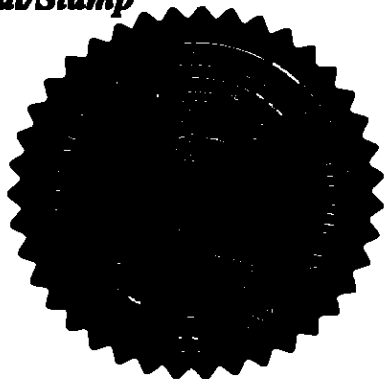
5 at Lansing, Michigan

6. the 6th of July, 2006

7 by Secretary of State, State of Michigan

8. NO 20589W-06

9. Seal/Stamp



10 Signature:

A handwritten signature in cursive script, reading "Terri Lynn Land".

Terri Lynn Land

This certification attests only to the authenticity of the signature of the official who signed the attested document, the capacity in which that official acted, and where appropriate, the identity of the seal or stamp which the document bears. This certification is not intended to imply that the contents of the document are correct, nor that they have the approval of the State of Michigan.

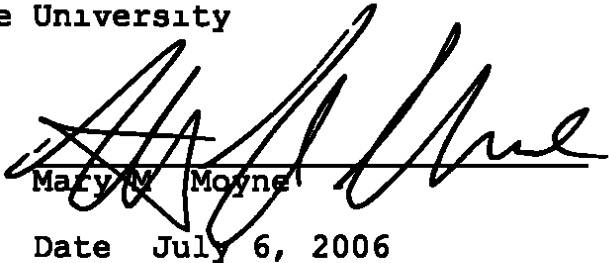
9 106

MSU 4 2-726 Colombia

TRUE COPY

Being duly sworn, I, MARY M MOYNE, an attorney at McLEOD & MOYNE, P C , having offices at 2190 Commons Parkway, Okemos, Michigan 48864, depose and say

THAT I have examined the attached photocopy of the original Assignment and have compared the copy with the original document and find that the attached document is a true and faithful reproduction of the original and purports to convey all right, title and interest in and to Colombian Patent Application No 06 042 613 entitled **STARCH-VEGETABLE OIL GRAFT COPOLYMERS AND THEIR BIOFIBER COMPOSITES, AND A PROCESS FOR THEIR MANUFACTURE** and to any and all Letters Patent to be obtained thereon to Board of Trustees of Michigan State University


Mary M Moyne
Date July 6, 2006

STATE OF MICHIGAN
COUNTY OF INGHAM ss

On this 6th day of July, 2006, the aforesaid MARY M MOYNE appeared before me and being duly sworn asserted the foregoing facts in regard to the attached documents are true to the best of her knowledge and belief


Notary Public

SEAL

My Commission Expires TAMMI L. TAYLOR
Notary Public, Ingham County, MI
My Commission Expires Oct. 27 2008



41-726
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102
IAN C MCLEOD

UNITED STATES PATENT AND TRADEMARK OFFICE

UNDER SECRETARY OF COMMERCE FOR INTELLECTUAL PROPERTY AND
DIRECTOR OF THE UNITED STATES PATENT AND TRADEMARK OFFICE

FEBRUARY 10, 2006

PTAS



103112184A

IAN C MCLEOD
MCLEOD & MOYNE, P C
2190 COMMONS PARKWAY
OKEMOS, MICHIGAN 48864

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RECORDATION DATE 10/26/2005

REEL/FRAME 017152/0430
NUMBER OF PAGES 5

BRIEF ASSIGNMENT OF ASSIGNOR'S INTEREST (SEE DOCUMENT FOR DETAILS)

ASSIGNOR

NARAYAN, RAMANI

DOC DATE 05/06/2005

ASSIGNOR

BALAKRISHNAN, SUNDER

DOC DATE 10/23/2005

ASSIGNOR

SHIN, BOO-YOUNG

DOC DATE 05/23/2005

ASSIGNEE

BOARD OF TRUSTEES OF MICHIGAN
STATE UNIVERSITY
238 ADMINISTRATION BUILDING
MICHIGAN STATE UNIVERSITY
EAST LANSING, MICHIGAN 48824

017152/0430 PAGE 2

SERIAL NUMBER 11124491

FILING DATE 05/06/2005

PATENT NUMBER

ISSUE DATE

TITLE STARCH-VEGETABLE OIL GRAFT COPOLYMERS AND THEIR BIOFIBER COMPOSITES,
AND A PROCESS FOR THEIR MANUFACTURE

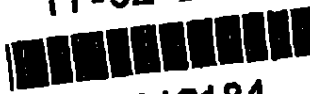
ALLYSON PURNELL, EXAMINER
ASSIGNMENT SERVICES BRANCH
PUBLIC RECORDS DIVISION

11-02-2005

Docket No. MSU 4.1-726

FORM PTO-1000 (Modified)
(Rev. 02-01)
OMB No. 0001-0027 (exp. 02/28/2002)
PDRREV03

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Patent and Trademark Office



103112184

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1 Name of conveying party(ies):

Ramesh Narayan
Sunder Balakrishnan
Bee-Young Sals

Additional name(s) of conveying party(ies)

☐ Yes ☒ No

3. Nature of conveyance:

☒ Assignment☐ Merger☐ Security Agreement☐ Change of Name☐ Other

R. Narayan 5/6/2005 S Balakrishnan
Execution Date: 10/23/2005 B. Shin 5/23/05

2. Name and address of receiving party(ies).

Name: Board of Trustees of Michigan State University

Internal Address:

Street Address: 238 Administration Building

MICHIGAN STATE UNIVERSITY

City East Lansing State: MI ZIP: 48824

Additional name(s) & address(es) attached? ☐ Yes ☒ No

4 Application number(s) or patent number(s):

If this document is being filed together with a new application, the execution date of the application is:

A. Patent Application No.(s)

11/124,491

B. Patent No (s)

Additional numbers attached? ☐ Yes ☒ No

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FINANCE SECTION

5. Name and address of party to whom correspondence concerning document should be mailed:

Name: Ian C. McLeod

Internal Address: McLeod & Moyne, P.C.

Street Address: 2190 Commons Parkway

City Okemos State: MI ZIP: 48864

6 Total number of applications and patents involved:

1

7 Total fee (37 CFR 3.41).

\$ 40.00

☒ Enclosed - Any excess or insufficiency should be credited or debited to deposit account☐ Authorized to be charged to deposit account

8 Deposit account number

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9. Statement and signature.

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Ian C. McLeod

Name of Person Signing

Signature

10/26/2005

Date

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1 Name of conveying party(ies)

Ramesh Narayan
Sunder Balakrishnan
Boo-Young Shin

Additional name(s) of conveying party(ies)

☐ Yes ☒ No

2 Name and address of receiving party(ies)

Name Board of Trustees of Michigan State University

Internal Address. _____

Street Address. 238 Administration BuildingMICHIGAN STATE UNIVERSITYCity East Lansing State: MI ZIP: 48824Additional name(s) & address(es) attached? ☐ Yes ☒ No

3 Nature of conveyance

☒ Assignment☐ Merger☐ Security Agreement☐ Change of Name☐ Other _____R. Narayan 5/6/2005 S. Balakrishnan
Execution Date 10/23/2005 B. Shin 5/23/05

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A. Patent Application No.(s)

11/124,491

B. Patent No.(s)

Additional numbers attached? ☐ Yes ☒ No

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Name Ian C. McLeodInternal Address: McLeod & Moyne, P.C.Street Address: 2190 Commons ParkwayCity Oakland State: MI ZIP: 48864

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1

7 Total fee (37 CFR 3.41)

\$ 40.00☒ Enclosed - Any excess or insufficiency should be credited or debited to deposit account☐ Authorized to be charged to deposit account

8 Deposit account number

13-0610

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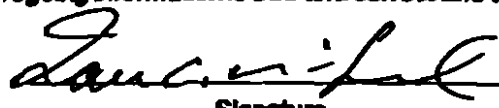
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To the best of my knowledge and belief, the foregoing information is true and correct and any attached copy is a true copy of the original document.

Ian C. McLeod

Name of Person Signing



Signature

10/26/2005

Date

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Director of the United States Patent and Trademark Office, P.O. Box 1450, Alexandria, VA 22313-1450

Go to Shaw
R. Narayan
10/26/05

ASSIGNMENT

WHEREAS, we, RAMANI NARAYAN, SUNDER BALAKRISHNAN and BOO-YOUNG SHIN, 4275 Conifer Circle, Okemos, Michigan 48864, 1636 Indian Way, Okemos, Michigan 48864, and Kyoungsan 712-749, Korea, respectively have invented certain new and useful improvements in STARCH-VEGETABLE OIL GRAFT COPOLYMERS AND THEIR BIOFIBER COMPOSITES, AND A PROCESS FOR THEIR MANUFACTURE for which an application for United States Letters Patent was filed on May 6, 2005 and assigned Serial No 11/124,491, and

WHEREAS, Board of Trustees, a Constitutional Corporation of MICHIGAN STATE UNIVERSITY, (hereinafter referred to as Assignee) of East Lansing, State of Michigan is desirous of acquiring the entire right, title and interest in and to said invention and in and to any Letters Patent that may be granted therefor in the United States and in any and all foreign countries

NOW, THEREFORE, in consideration of the sum of One Dollar (\$1 00) to us in hand paid, the receipt whereof is hereby acknowledged, and other valuable considerations, we the said RAMANI NARAYAN, SUNDER BALAKRISHNAN, AND BOO-YOUNG SHIN, have sold, assigned and transferred, and by these presents do sell, assign and transfer unto said Assignee the full and exclusive right to the said invention in the United States and its territorial possessions and in all foreign countries and the entire right, title and interest in and to any and all Letters Patent which may be granted therefor in the United States and its territorial possessions and in any and all foreign countries and in and to any and all divisions, reissues, continuations and extensions thereof

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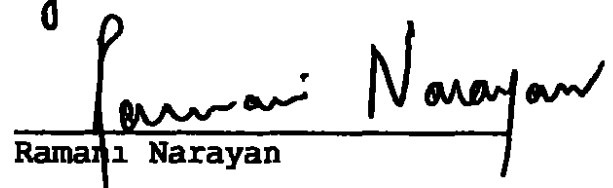
We hereby authorize and request the Patent and Trademark Office officials in the United States and any and all foreign countries to issue any and all of said Letters Patent, when granted, to said Assignee as the Assignee of our entire right, title and interest in and to the same for the sole use and behoof of the said Assignee, its successors and assigns

FURTHER, we agree that we will communicate to said Assignee or its representatives any facts known to us respecting said invention, and testify in any legal proceeding, sign all lawful papers, execute all divisional, continuation, substitution, renewal and reissue applications, execute all necessary assignment papers to cause any and all of said Letters Patent to be issued to said Assignee, make all rightful oaths and generally do everything possible to aid said Assignee, its successors and assigns, to obtain and enforce proper protection for said invention in the United States and in any and all foreign countries

IN TESTIMONY WHEREOF, I have hereunto set my hand this 6th day of May, 2005
Signed in the Presence of



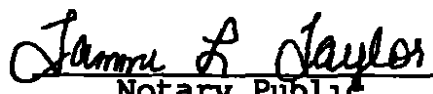
Ramani Narayan



Ramani Narayan

STATE OF MICHIGAN
COUNTY OF INGHAM

On this 6th day of May, 2005, personally appeared before me the above named Ramani Narayan to me known and known to me to be the person described in and who executed the foregoing instrument and acknowledged the same to be his free act and deed in and for the purposes set forth in said instrument.

SEAL


Notary Public

My Commission Expires _____ TAMMIL TAYLOR
Notary Public Ingham County MI
My Commission Expires Oct 27 2008

113

IN TESTIMONY WHEREOF, I have hereunto set my
hand this 23rd day of October, 2005

Signed in the Presence of

Darcie L. Raynor

Sunder Balakrishnan
Sunder Balakrishnan

STATE OF MICHIGAN

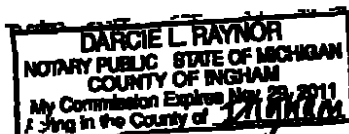
ss

COUNTY OF INGHAM

On this 23rd day of OCTOBER, 2005,
personally appeared before me the above named Sunder
Balakrishnan to me known and known to me to be the
person described in and who executed the foregoing
instrument and acknowledged the same to be his free act
and deed in and for the purposes set forth in said
instrument

SEAL

Darcie L. Raynor
Notary Public
My Commission Expires Nov 29, 2011



IN TESTIMONY WHEREOF, I have hereunto set my

hand this 23rd day of May, 2005

Signed in the Presence of

Boo-Young Shin

Boo-Young Shin
Boo-Young Shin

CITY OF

ss

COUNTRY OF

On this 23TH day of MAY, 2005,
personally appeared before me the above named Boo-Young
Shin to me known and known to me to be the person
described in and who executed the foregoing instrument
and acknowledged the same to be his free act and deed in
and for the purposes set forth in said instrument

SEAL

Boo-Young Shin
Notary Public

My Commission Expires YOUNGNAM LAW OFFICE
33-2 BUMED-DONG SUSUNG-GU DAEGU, KOREA
THIS OFFICE HAS BEEN AUTHORIZED BY THE MINISTER
OF JUSTICE, THE REPUBLIC OF KOREA, TO ACT AS
NOTARY PUBLIC SINCE SEP 27TH, 1993 UNDER LAW
NO 3594

14 774

등부 2005 년 제 1430 호

Registered No 2005 - 1430

인증

Notarial Certificate

위 양 도 중

Boo-Young Shun

에 기재된

personally appeared before me

신 부 영 은

and admitted his (her)

본직의 면전에서 위사서증서에 자기가
서명날인한 것임을 자인하였다

subscription to the attached

Assignment

2005 년 5 월 23 일 이 사무소에서
위 인증한다

This is hereby attested on
this 23th day of May, 2005
at this office



공증인가 영남법무법인

대구광역시 수성구 범어동 33-2

YOUNGNAM LAW OFFICE

33-2 Bumed-dong susung-gu

DAEGU, KOREA

공증담당 변호사



Attorney - at - Law

This office has been
authorized by the Minister of
Justice, the Republic of
Korea, to act as Notary
Public since

sep 27th, 1993

under Law No 3594

공증인가 영남법무법인 전화 : 743-7584, 7585

18 115

**TRADUCCION AL ESPAÑOL del Documento de Cesión a MICHIGAN
STATE UNIVERSITY firmado por los cedentes y el cesionario**

ESTADO DE MICHIGAN

DEPARTAMENTO DE ESTADO

**APOSTILLA
(Convención de La Haya del 5 de octubre de 1961)**

1 País ESTADOS UNIDOS DE AMÉRICA

Este documento público
2 ha sido firmado por Tammi L Taylor
3 actuando en capacidad de Notario publico de Michigan
4 lleva el sello de Tammi L Taylor
Condado de Ingham, Michigan

CERTIFICADO

5 en Lansing, Michigan 6 el 6 de julio de 2006
7 por el Secretario de Estado, Estado de Michigan
8 NO 20589W-06
9 Sello/timbre 10 Firma

(Firma)
Tern Lynn Land

La certificación da testimonio de la autenticidad de la firma del funcionario que firmó el documento anexo, la capacidad en la cual actuó ese funcionario, y donde sea apropiado, la identidad del sello o timbre que lleva el documento. Esta certificación no tiene intención de implicar que los contenidos del documento son correctos, ni que tienen la aprobación del Estado de Michigan

116

MSU 4 2-726 Colombia

COPIA VERDADERA

Estando debidamente jurado, yo, **MARY M MOYNE**, un abogado en **McLEOD & MOYNE, P C**, teniendo oficinas en **2190 Commons Parkwat, Okemos, Michigan 48864**, declaro y digo

QUE he examinado la fotocopia anexa de la cesión original y he comparado la copia con el documento original y encontré que el documento anexo es una reproducción fiel y verdadera del original y tiene el propósito de ceder todo el derecho, título e interés en y para la solicitud colombiana de patente No 06 042 316 trtutada **COPOLIMEROS INJERTADOS DE ALMIDÓN-ACEITE VEGETAL Y SUS COMPUESTOS DE BIOFIBRA, Y UN PROCESO PARA SU FABRICACIÓN** y para cualquiera y todas las patentes que se obtenga sobre ella a Board Of Trustees of Michigan State University

(Firma)

Mary M Moyne

Fecha Julio 6 de 2006

ESTADO DE MICHIGAN

ss

CONDADO DE INGHAM

Este 6 de julio de 2006, la amba mencionada **MARY M MOYNE** apareció ante mí y estando debidamente jurada asevero los anteriores hechos respecto a los documentos anexos que son ciertos hasta donde sabe y cree

(Firma)

Notario publico

[Sello]

Mi comisión expira

Hay un sello que dice

TAMMI L TAYLOR

Notario publico, Condado de Ingham, MI

Mi comisión expira Octubre 27 de 2008

20 177

FEBRERO 10 DE 2006

IAN C MCCLEOD
MCLEOD & MOYNE P C
2190 COMMONS PARKWAY
OKEMOS, MICHIGAN 48864

OFICINA DE MARCAS Y PATENTES DE LOS ESTADOS UNIDOS
INFORME DE GRABACIÓN DE DOCUMENTO DE CESION

EL DOCUMENTO ADJUNTO HA SIDO GRABADO POR LA DIVISIÓN DE CESIONES DE LA OFICINA DE MARCAS Y PATENTES DE LOS ESTADOS UNIDOS HAY DISPONIBLE UNA COPIA COMPLETA EN MICROFILME EN EL CUARTE DE BUSQUEDA DE CESIONES EN EL NUMERO DE ROLLO Y CUADRO REFERENCIADOS ABAJO

POR FAVOR REVISE TODA LA INFORMACIÓN CONTENIDA EN ESTE INFORME LA INFORMACIÓN CONTENIDA EN ESTE INFORME DE GRABACIÓN REFLEJA LOS DATOS PRESENTES EN EL SISTEMA DE ASIGNACIÓN DE PATENTES Y MARCAS SI USTED ENCUENTRA CUALQUIER ERROR O TIENE PREGUNTAS CONCERNIENTES A ESTE INFORME PUEDE CONTACTAR AL EMPLEADO CUYO NOMBRE APARECE EN ESTE INFORME AL 703-308-8723 POR FAVOR ENVÍE LA SOLICITUD DE CORRECCIÓN A OFICINA DE MARCAS Y PATENTES DE LOS ESTADOS UNIDOS, DIVISIÓN DE CESIONES, ASIGNACIÓN DE CASILLAS, CG-4, 1213 JEFFERSON DAVIS HWY, SUITE 320, WASHINGTON, D C 20231

FECHA DE GRABACIÓN	10/26/2005	ROLLO/CUADRO	017152/0430
		NUMERO DE PÁGINAS	5

RESUMEN CESIÓN DE INTERÉS DEL CEDENTE (VER DOCUMENTO PARA DETALLES)

CEDENTE		FECHA DE DOC	05/06/2005
NARAYAN RAMANI			

CEDENTE		FECHA DE DOC	10/23/2005
BALAKRISHNAN, SUNDER			

CEDENTE		FECHA DE DOC	05/25/2005
SHIN, BOO-YOUNG			

CESIONARIO

BOARD OF TRUSTEES OF MICHIGAN
STATE UNIVERSITY
238 ADMINISTRATION BUILDING
MICHIGAN STATE UNIVERSITY
EAST LANSIG, MICHIGAN 48824

2/ 178

017152/0430

NUMERO DE SERIE 11124491

FECHA DE PRESENTACIÓN 05/08/2005

NUMERO DE PATENTE

FECHA DE EMISIÓN

TITULO COPOLIMEROS INJERTADOS DE ALMIDON-ACEITE VEGETAL Y SUS
COMPUESTOS DE BIOFIBRA, Y UN PROCESO PARA SU FABRICACION

ALISTON PURNELL EXAMINADOR
DIVISIÓN DE CESIONES
OFICINA DE REGISTROS PUBLICOS

/





FORMULARIO PTO-1585 (Rev. 8-93) OMB No. 0651-0011 (exp. 4/94) TU0059 (PSP)		PORTADA DEL FORMULARIO DE REGISTRO 700026737 SOLO PATENTES		Departamento de Comercio de los Estados Unidos Oficina de Marcas y Patentes				
Al Honorable Comisionado de Marcas y Patentes por favor registre los documentos originales adjuntos o copias de los mismos								
1 Nombre de la(s) parte(s) cedente(s) Ramani Narayan Sunder Balakrishnan Boo-Young Shin Se adjunta(n) nombre(s) adicional(es) de la(s) parte(s) cedente(s) ☐ SI ☒ No			2 Nombre y dirección de la(s) parte(s) que recibe(n) Nombre <u>Board of Trustees of Michigan State University</u> Dirección Interna _____ Dirección <u>238 Administration Building</u> <u>MICHIGAN STATE UNIVERSITY</u> Ciudad <u>East Lansing</u> Estado <u>MI</u> ZIP <u>48824</u> ¿Se adjunta(n) nombre(s) y dirección(es) adicional(es)? ☐ SI ☒ No					
3 Naturaleza del traspaso ☒ Cesión ☐ Fusión ☐ Acuerdo de Seguridad ☐ Cambio de Nombre ☐ Otro _____ Fecha de Ejecución <u>R Naryanan 5/16/2005 S Balakrishnan 10/23/2005 B Shin 5/23/05</u>			4 Número(s) de Solicitud o número(s) de Patente Si este documento está siendo entregado junto con una nueva solicitud la fecha de ejecución de la solicitud es. _____ <table style="width: 100%;"> <tr> <td style="width: 50%; vertical-align: top;"> A Solicitud(es) de Patente No(s) <u>11/124 491</u> </td> <td style="width: 50%; vertical-align: top;"> B No(s) de Patente </td> </tr> </table> ¿Se adjuntan números adicionales? ☐ SI ☒ No			A Solicitud(es) de Patente No(s) <u>11/124 491</u>	B No(s) de Patente 	
A Solicitud(es) de Patente No(s) <u>11/124 491</u>	B No(s) de Patente 							
5 Nombre y dirección de la parte a quien se le debe enviar la correspondencia de los documentos concernientes Nombre <u>Ian C. McLeod</u> Dirección Interna <u>McLeod & Moyne, P.C.</u> Dirección <u>2190 Commons Parkway</u> Ciudad <u>Okemos</u> Estado <u>MI</u> ZIP <u>48864</u>			6 Número total de solicitudes y patentes involucradas <u>1</u> 7 Tarifa Total (37 CFR 3.41) \$ <u>40</u> ☒ Adjunta ☐ Autorización para ser cargada a una cuenta de depósito y cualquier otra tarifa adicional requerida 8 Número de cuenta depósito <u>13-0610</u> (Anexar copia por duplicado de esta página al se paga por cuenta de depósito)					
NO USAR ESTE ESPACIO								
9 Declaración y firma Hasta donde se y creo la información anterior es veraz y correcta y cualquier copia adjunta es una copia veraz del documento original <table style="width: 100%;"> <tr> <td style="width: 30%;"> <u>Ian C. McLeod</u> Nombre del suscrito </td> <td style="width: 30%; text-align: center;"> _____ (Firma) Firma </td> <td style="width: 30%; text-align: center;"> _____ 10/26/2005 Fecha </td> </tr> </table> ☐ Certificado de envío por el reverso						<u>Ian C. McLeod</u> Nombre del suscrito	_____ (Firma) Firma	_____ 10/26/2005 Fecha
<u>Ian C. McLeod</u> Nombre del suscrito	_____ (Firma) Firma	_____ 10/26/2005 Fecha						
Número total de páginas incluyendo la portada anexos y documentos 5								

29 120

FORMULARIO PTO-1595 (Rev 8-93) OMB No 0851-0011 (exp 4/94) TU0059 (PSP)		PORTADA DEL FORMULARIO DE REGISTRO 700026737 SOLO PATENTES		Departamento de Comercio de los Estados Unidos Oficina de Marcas y Patentes	
Al Honorable Comisionado de Marcas y Patentes por favor registre los documentos originales adjuntos o copias de los mismos					
10 Nombre de la(s) parte(s) cedente(s) Raman Narayan Sunder Balakrishnan Boo-Young Shin Se adjunta(n) nombre(s) adicional(es) de la(s) parte(s) cedente(s) ☐ SI ☒ No			11 Nombre y dirección de la(s) parte(s) que recibe(n) Nombre <u>Board of Trustees of Michigan State University</u> Dirección Interna _____ Dirección <u>238 Administration Building</u> <u>MICHIGAN STATE UNIVERSITY</u> Ciudad <u>East Lansing</u> Estado <u>MI</u> ZIP <u>48824</u> ¿Se adjunta(n) nombre(s) y dirección(es) adicional(es)? ☐ SI ☒ No		
12 Naturaleza del traspaso ☒ Cesión ☐ Fusión ☐ Acuerdo de Seguridad ☐ Cambio de Nombre ☐ Otro _____ Fecha de Ejecución R Naryanan 5/16/2005 S Balakrishnan 10/23/2005 B Shin 5/23/05					
13 Número(s) de Solicitud o número(s) de Patente Si este documento está siendo entregado junto con una nueva solicitud la fecha de ejecución de la solicitud es _____ C Solicitud(es) de Patente No(s) 11/124 491 D No(s) de Patente _____ ¿Se adjuntan números adicionales? ☐ SI ☒ No					
14 Nombre y dirección de la parte a quien se le debe enviar la correspondencia de los documentos concernientes Nombre <u>Ian C. McLeod</u> Dirección Interna <u>McLeod & Moyne, P C</u> Dirección <u>2180 Commons Parkway</u> Ciudad <u>Okemos</u> Estado <u>MI</u> ZIP <u>48864</u>			15 Número total de solicitudes y patentes involucradas <u>1</u> 16 Tarifa Total (37 CFR 3.41) \$ <u>40</u> ☒ Adjunta ☐ Autorización para ser cargada a una cuenta de depósito y cualquier otra tarifa adicional requerida 17 Número de cuenta depósito <u>13-0610</u> (Anexar copia por duplicado de esta página si se paga por cuenta de depósito)		
NO USAR ESTE ESPACIO					
18 Declaración y firma Hasta donde se y creo la información anterior es veraz y correcta y cualquier copia adjunta es una copia veraz del documento original Ian C. McLeod _____ (Firma) _____ 10/26/2005 Nombre del suscrito Firma Fecha ☐ Certificado de envío por el reverso Número total de páginas incluyendo la portada anexos y documentos 5					

24 121

MSU 4 1-726

CESION

CONSIDERANDO QUE, nosotros, RAMANI NARAYAN, SUNDER BALAKRISHAN y BOO-YOUNG SHIN, 4275 Conifer Chicle, Okemos, Michigan 48864, 1636 Indian Way, Okemos, Michigan 48864, y Kyoungsan 712-749, Corea, respectivamente hemos inventado ciertas mejoras nuevas y utiles en COPOLIMEROS INJERTADIS DE ALMIDÓN-ACEITE VEGETAL Y SUS COMPUESTOS DE BIOFRIBRA, Y UN PORCESO PARA SU FABRICACION para las cuales se presentó solicitud de patente de Estados Unidos el 6 de mayo de 2005 y se le asignó el numero de sene 11/124,491, y

CONSIDERANDO QUE, Board of Trustees, una Corporación Constitucional de MICHIGAN STATE UNIVERSITY, (en adelante referida como el Cesionario) de East Lansing, Estado de Michigan esta deseosa de adquirir la totalidad del derecho título e interés en y para dicha invención y en y para todas las patentes que le puedan ser concedidas en Estados Unidos y en cualquiera y todos los países extranjeros

AHORA, POR LO TANTO, en consideración de la suma de un dólar (\$1 00) que se nos pagó en la mano, el recibo del cual es reconocido aquí, y otras consideraciones valiosas, nosotros los dichos RAMANI NARAYAN, SUNDER BALAKRISHAN y BOO-YOUNG SHIN, hemos vendido, cedido y transfendo, y por la presente vendemos, cedemos y transferimos a dicho Cesionario el derecho total y exclusivo a dicha invención en Estados Unidos y sus posesiones territoriales y en todos los países extranjeros y la totalidad del derecho, título e interés en y para cualquier y todas las patentes que sean concedidas a la misma en Estados Unidos y sus posesiones territoriales y en cualquiera y todos los países extranjeros y en y para cualquiera y todas las divisiones, reemisiones, continuaciones y extensiones de la misma

Por la presente autorizamos y solicitamos a los funcionarios de la oficina de marcas y patentes en Estados Unidos y en cualquiera y todos los países extranjeros a emitir cualquiera y todas las dichas patentes, cuando se concedan, a dicho cesionario como el cesionario de la totalidad de nuestro derecho, título e interés en y para la misma para el uso y disfrute único de dicho cesionario, sus sucesores o cesionarios

ADEMÁS, acordamos que comunicaremos a dicho cesionario o sus representantes cualquier hecho conocido por nosotros respecto a dicha invención, y testificaremos en cualquier proceso legal, firmaremos cualquier documento legal, ejecutaremos todas solicitudes de división, continuación, sustitución y reemisión, ejecutaremos todos los documentos de cesión para ocasionar que cualquiera y todas las dichas patentes sean emitidas al cesionario, haremos todos los juramentos debidos y generalmente haremos todo lo posible para ayudar a dicho cesionario, sus sucesores o cesionarios, a obtener y ejercer la protección apropiada para dicha invención en Estados Unidos y cualquiera y todos los países extranjeros

EN TESTIMONIO DE LO CUAL, he puesto mi firma al presente este 6° día de mayo de 2005
Firmado en presencia de

(Firma)

(Firma)
Ramani Narayan

ESTADO DE MICHIGAN

ss

CONDADO DE INGHAM

En este 6° día de mayo de 2005, personalmente apareció ante mí el arriba nombrado Ramani Narayan a quien conozco y reconozco como la persona descrita en y quien ejecutó el anterior instrumento y lo reconoció como su acto libre y título en y para los propósitos establecidos en dicho instrumento

Sello

(Firma)
Notario publico
Mi comisión expira TAMMI L. TAYLOR
Notario público, Condado de Ingham, MI
Mi comisión expira Oct 27 de 2008

24 123

EN TESTIMONIO DE LO CUAL, he puesto mi firma al presente este
23^{er} día de octubre de 2005
Firmado en presencia de

(Firma)

(Firma)
Sunder Balakrishnan

ESTADO DE MICHIGAN

ss

CONDADO DE INGHAM

En este 23^{er} día de octubre de 2005, personalmente apareció ante mí el arriba nombrado Sunder Balakrishnan a quien conozco y reconozco como la persona descrita en y quien ejecutó el anterior instrumento y lo reconoció como su acto libre y título en y para los propósitos establecidos en dicho instrumento

Sello

(Firma)
Notario público

Mi comisión expira Nov 29 de 2011

DARCIE L RAYNOR
NOTARIO PUBLICO – ESTADO DE MICHIGAN
CONDADO DE INGHAM
Mi comisión expira Nov 29 de 2011
Actuando en el condado de Ingham

EN TESTIMONIO DE LO CUAL, he puesto mi firma al presente este
23^{er} día de mayo de 2005
Firmado en presencia de

(Firma)

(Firma)
Boo-Young Shin

CIUDAD

ss

PAÍS

En este 23^{er} día de mayo de 2005, personalmente apareció ante mí el arriba nombrado Boo-Young Shin a quien conozco y reconozco como la persona descrita en y quien ejecutó el anterior instrumento y lo reconoció como su acto libre y título en y para los propósitos establecidos en dicho instrumento

Sello

(Firma)
Notario publico

Mi comisión expira _____

YOUNGMAN LAW OFFICE
33-2 BUMED-DONG SUSUNG-GU DAEGU, COREA
ESTE OFICINA HA SIDO AUTORIZADA POR EL MINISTRO
DE JUSTICIA, LA REPUBLICA DE COREA, PARA ACTUAR COMO
NOTARIO PUBLICO DESDE SEPT 27 DE 1993, BAJO LA LEY NO 3594

22

124

Registro No 2005 – 1430

Certificado Notanal

Boo-Young Shin

apareció personalmente ante mi

y admitió su

suscnpción al anexo

Cesión

Esto se certfica este

23^{er} día de mayo de 2005

en esta oficina

YOUNGNBAM LAW OFFICE

33-2 Burned-dong susung-gu

DAEGU, COREA

(Firma)

Esta oficina ha sido

autonzada por el Ministerio de

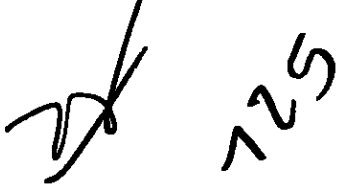
Justicia, Republica de

Corea, para actuar como notano

publico desde

Sept 27 de 1993

BAJO LA LEY No 3594


**TRADUCCIÓN DEL INGLÉS AL ESPAÑOL de la Publicación de la Solicitud de
Patente de los Estados Unidos de América No 11/124,491 relacionada con**

**COPOLIMEROS INJERTADOS DE ACEITE VEGETAL – ALMIDON Y SUS
COMPUESTOS DE BIOFIBRA, Y UN PROCESO PARA SU FABRICACION**

**ANEXOS La Publicación de la Solicitud de Patente de los Estados Unidos No
11/124,491 arriba mencionada y Certificación del Comisionado de Patentes y
Marcas de los Estados Unidos**

**LOS ESTADOS UNIDOS DE AMÉRICA
A TODO AQUEL A QUIEN PUEDA LLEGARLE LO PRESENTE**

**DEPARTAMENTO DE COMERCIO DE ESTADOS UNIDOS
Oficina de Marcas y Patentes de Estados Unidos**

Junio 23 de 2006

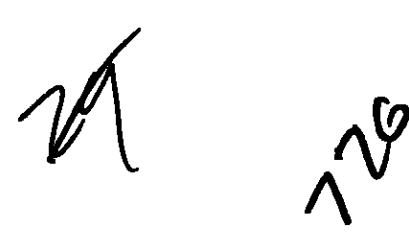
**ESTE ES PARA CERTIFICAR QUE ANEXO A LA PRESENTE HAY UNA COPIA
VREDADERA DE LOS ARCHIVOS DE LA OFICINA DE MARCAS Y PATENTES
DE ESTADOS UNIDOS DE AQUELLOS DOCUMENTOS DE LA SOLICITUD DE
PATENTE IDENTIFICADA DEBAJO QUE CUMPLE LOS REQUERIMIENTOS
PARA QUE SE LE CONCEDA UNA FECHA DE PRESENTACION BAJO EL 35
USC 111**

**Número de publicación de solicitud de patente 11/124,491
Fecha de publicación Mayo 06 de 2005**

**EL CODIGO DEL PAIS Y NUMERO DE SU SOLICITUD DE PRIORIDAD, A SER
UTILIZADO PARA PRESENTACIONES EN EL EXTRANJERO BAJO LA
CONVENCION DE PARIS, ES US11/124,491**

**Por autoridad del
Subsecretario de Comercio para Propiedad Intelectual
y Director de la Oficina de Marcas y Patentes de Estados Unidos**

**(Firma)
L EDELEN
Funcionario que certifica**



NOSOTROS REIVINDICAMOS

1 Una composición de copolímero biodegradable de almidón-acerte vegetal la cual es una mezcla combinada reactivamente que comprende un almidón granular, un aceite vegetal o derivado del mismo que comprende un modificador reactivo con el almidón, y un plastificante hidroxilado

2 La composición de la reivindicación 1 en donde el almidón granulado se selecciona del grupo que consiste de almidón de maíz, almidón de papa, almidón de tapioca, almidón de arroz, almidón de trigo, almidón de casave y almidón de maíz con alto contenido de amilasa

3 La composición de la reivindicación 1 en donde el almidón en la mezcla es 5% a 90% en peso

4 La composición de la reivindicación 1, en donde el almidón tiene un contenido de humedad de alrededor de 0 5% a 15% en peso del almidón

5 La composición de la reivindicación 1 en donde el almidón está en forma pulverizada

6 La composición de la reivindicación 1 en donde el almidón es modificado químicamente con un modificador

7 La composición de la reivindicación 1 en donde el aceite vegetal es un derivado el cual es epoxilado, hidroxilado o maleatado

8 La composición de la reivindicación 1 en donde el aceite vegetal se selecciona del grupo que consiste de aceite de maíz, aceite de soya, aceite de soya maleatado y aceite de soya epoxilado y mezclas de estos

9 La composición de la reivindicación 1 en donde el aceite vegetal es 1% a 10% en peso de la composición

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10 La composición de la reivindicación 1 en donde el plastificante es un alcohol polihídrico en una cantidad entre 10 y 40 % en peso del total de la composición

11 La composición de la reivindicación 1 en donde se proporciona un iniciador de peróxido de radicales libres en la mezcla

12 La composición de la reivindicación 11 en donde el iniciador de radicales libres es 2,5-Bis(tert-butilperoxi)-2,5-dimetilhexano (LUPEROX 101)

13 La composición de la reivindicación 11 en donde se proporciona un iniciador de peróxido de radicales libres en la mezcla en una cantidad de entre alrededor de 0.01 a 3% en peso del peso total de la composición

14 La composición de la reivindicación 1 que comprende además fibra

15 La composición de la reivindicación 14 en donde la fibra se selecciona del grupo que consiste de algodón, madera, lino, ábaca, ramio, cáñamo, y bagazo

16 La composición de la reivindicación 15 en donde la fibra tiene una proporción de aspecto de al menos alrededor de 10:1

17 La composición de la reivindicación 12 en donde la fibra está presente en una cantidad entre alrededor de 3% a 80% en peso de la composición

18 La composición de la reivindicación 1 que comprende el modificador el cual se selecciona del grupo que consiste de ácido cíclico o dibásico y anhídridos del mismo

19 La composición de la reivindicación 18 en donde el modificador está presente en la composición en una cantidad desde alrededor de 0.1 a alrededor de 10% en peso del almidón

20 La composición de la reivindicación 1 que comprende además un poliéster biodegradable seleccionado del grupo que consiste de

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poli(carbolactona)(PCL), poli(vinilacetato – co – vinyl alcohol) (PVAc/VA), poli(ácido láctico) o poliláctido (PLA), poli(ácido glicólico) o poliglicólido (PGA), poli(beta hidroxialcanoatos) (PHA), y copoliésteres alifáticos aromáticos

21 La composición de la reivindicación 20 en donde el poliéster biodegradable es 1% a 60% en peso de la composición

22 Un proceso para producir una composición de copolímero de almidón y aceite vegetal biodegradable el cual comprende

(a) combinar reactivamente una mezcla de un almidón granular, un aceite vegetal o un derivado del mismo que comprende un modificador reactivo con el almidón, y un plastificante en un reactor, y

(b) remover la composición del reactor para producir la composición

23 El proceso de la reivindicación 22 en donde el reactor es un extrusor

24 El proceso de la reivindicación 22 en donde el derivado de aceite vegetal es un aceite vegetal maleatado

25 El proceso de la reivindicación 22 en donde la mezcla comprende además una fibra

26 El proceso de la reivindicación 22 en donde el plastificante es un alcohol polihídrico

DA 1482813

**THE UNITED STATES OF AMERICA****TO ALL TO WHOM THESE PRESENTS SHALL COME:****UNITED STATES DEPARTMENT OF COMMERCE****United States Patent and Trademark Office****June 23, 2006**

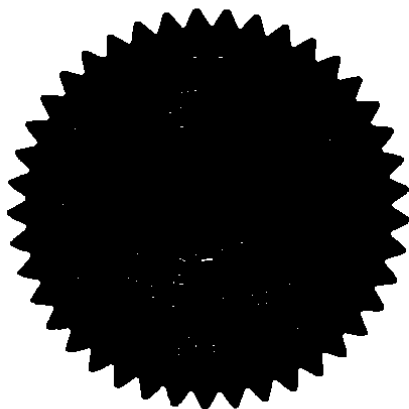
**THIS IS TO CERTIFY THAT ANNEXED HERETO IS A TRUE COPY FROM
THE RECORDS OF THE UNITED STATES PATENT AND TRADEMARK
OFFICE OF THOSE PAPERS OF THE BELOW IDENTIFIED PATENT
APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A
FILING DATE UNDER 35 USC 111**

APPLICATION NUMBER 11/124,491**FILING DATE May 06, 2005**

**THE COUNTRY CODE AND NUMBER OF YOUR PRIORITY
APPLICATION, TO BE USED FOR FILING ABROAD UNDER THE PARIS
CONVENTION, IS US11/124,491**

By Authority of the**Under Secretary of Commerce for Intellectual Property
and Director of the United States Patent and Trademark Office**

**L. EDELEN
Certifying Officer**



Preliminary Classification.
Proposed Class.
Subclass

NOTE: "All applicants are requested to include a preliminary classification on newly filed patent applications. The preliminary classification, preferably class and subclass designations, should be identified in the upper right-hand corner of the letter of transmittal accompanying the application papers, for example 'Proposed Class 2, subclass 123.' " M.P.E.P. § 801 8th ed.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Commissioner for Patents
P.O. Box 1450
Alexandria VA 22313-1450

NEW APPLICATION TRANSMITTAL

Transmitted herewith for filing is the patent application of

Inventor(s): Ramani Narayan, Sunder Balakrishnan and Boo-Young Shin

WARNING: 37 C.F.R. § 1.41(a)(1) points out:

"(a) A patent is applied for in the name or names of the actual inventor or inventors.

"(1) The inventorship of a nonprovisional application is that inventorship set forth in the oath or declaration as prescribed by § 1.63, except as provided for in § 1.53(c)(4) and § 1.63(c). If an oath or declaration as prescribed by § 1.63 is not filed during the pendency of a nonprovisional application, the inventorship is that inventorship set forth in the application papers filed pursuant to § 1.53(c), unless a petition under this paragraph accompanied by the fee set forth in § 1.17(i) is filed supplying or changing the name or names of the inventor or inventors." For (title).

**STARCH-VEGETABLE OIL GRAFT COPOLYMERS AND THEIR BIOFIBER COMPOSITES,
AND A PROCESS FOR THEIR MANUFACTURE**

EXPRESS MAILING UNDER 37 C.F.R. § 1.10*

(Express Mail label number is mandatory.)

(Express Mail certification is optional.)

I hereby certify that this paper, along with any document referred to, is being deposited with the United States Postal Service on this date May 6, 2005 in an envelope addressed to the Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 as "Express Mail Post Office to Addressee" Mailing Label No. ED 787639368 US

Tammi L. Taylor

(Type or print name of person mailing paper)

Tammi L. Taylor

Signature of person certifying

WARNING: Certificate of mailing (first class) or facsimile transmission procedures of 37 C.F.R. 1.8 cannot be used to obtain a date of mailing or transmission for this correspondence.

*WARNING: Each paper or fee filed by "Express Mail" must have the number of the "Express Mail" mailing label placed thereon prior to mailing. 37 C.F.R. 1.10(b).

"Since the filing of correspondence under § 1.10 without the Express Mail mailing label thereon is an oversight that can be avoided by the exercise of reasonable care, requests for waiver of this requirement will not be granted on petition." Notice of Oct. 24, 1992, 59 Fed. Reg. 55,439, at 55,442.

(New Application Transmittal [4-1]—page 1 of 1)

Best Available Copy

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1 Type of Application

This new application is for a(n)

(check one applicable item below)

- ☒ Original (nonprovisional)
☐ Design
☐ Plant

WARNING: Do not use this transmittal for a completion in the U.S. of an International Application under 35 U.S.C. § 371(c)(4), unless the International Application is being filed as a divisional, continuation or continuation-in-part application.

WARNING: Do not use this transmittal for the filing of a provisional application.

NOTE: If one of the following 3 items apply, then complete and attach **ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF A PRIOR U.S. APPLICATION CLAIMED** and a **NOTIFICATION IN PARENT APPLICATION OF THE FILING OF THIS CONTINUATION APPLICATION**.

- ☐ Divisional
☐ Continuation.
☐ Continuation-in-part (C-I-P)

2. Benefit of Prior U.S. Application(s) (35 U.S.C. §§ 110(a), 120, or 121)

NOTE: "A nonprovisional application or international application designating the United States of America may claim an invention disclosed in one or more prior-filed depending nonprovisional applications or international applications designating the United States of America. In order for an application to claim the benefit of a prior-filed depending nonprovisional application or international application designating the United States of America, each prior-filed application must name as an inventor at least one inventor named in the later-filed application and disclose the named inventor's invention claimed in at least one claim of the later-filed application in the manner provided by the first paragraph of 35 U.S.C. 112. In addition, each prior-filed application must be:

(i) An international application entitled to a filing date in accordance with PCT Article 11 and designating the United States of America, or

(ii) Complete as set forth in § 1.51(b), or

(iii) Entitled to a filing date as set forth in § 1.53(b) or § 1.53(d) and include the basic filing fee set forth in § 1.16; or

(iv) Entitled to a filing date as set forth in § 1.53(b) and have paid therein the processing and retention fee set forth in § 1.21(f) within the time period set forth in § 1.53(f).

37 C.F.R. § 1.78(h)(1).

WARNING: If an application claims the benefit of the filing date of an earlier filed application under 35 U.S.C. §§ 120, 121 or 365(c), the 20-year term of that application will be based upon the filing date of the earliest U.S. application that the application makes reference to under 35 U.S.C. §§ 120, 121 or 365(c). (35 U.S.C. § 154(b)(2) does not take into account, for the determination of the patent term, any application on which priority is claimed under 35 U.S.C. §§ 110, 365(b) or 365(c).) For a c-i-p application, applicant should review whether any claim in the patent that will issue is supported by an earlier application and, if not, the applicant should consider canceling the reference to the earlier filed application. The term of a patent is not based on a claim-by-claim approach. See Notice of April 14, 1995, 60 Fed. Reg. 20,195, at 20,505.

(New Application Transmittal [4-1]—page 2 of 16)

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WARNING: 37 C.F.R. § 1.76(b)(2) deals with the time in which the claim for the benefit of an earlier filing date must be made and states:

"(2)(f) Except for a continued prosecution application filed under § 1.53(c), any nonprovisional application or international application designating the United States of America claiming the benefit of one or more prior-filed copending nonprovisional applications or international applications designating the United States of America must contain or be amended to contain a reference to each such prior-filed application, identifying it by application number (consisting of the series code and serial number) or international application number and international filing date and indicating the relationship of the applications. Cross references to other related applications may be made when appropriate (see § 1.14).

(g) This reference must be submitted during the pendency of the later-filed application. If the later-filed application is an application filed under 35 U.S.C. 111(a), this reference must also be submitted within the later of four months from the actual filing date of the later-filed application or sixteen months from the filing date of the prior-filed application. If the later-filed application is a nonprovisional application which entered the national stage from an international application after compliance with 35 U.S.C. 371, this reference must also be submitted within the later of four months from the date on which the national stage commenced under 35 U.S.C. 371(a) or (i) in the later-filed international application or sixteen months from the filing date of the prior-filed application. These time periods are not extendable. Except as provided in paragraph (h)(2) of this section, the failure to timely submit the reference required by 35 U.S.C. 120 and paragraph (h)(2)(i) of this section is considered a waiver of any benefit under 35 U.S.C. 120, 121, or 368(c) to such prior-filed application. The time periods in this paragraph do not apply if the later-filed application is:

(A) An application for a design patent;

(B) An application filed under 35 U.S.C. 111(a) before November 29, 2000; or

(C) A nonprovisional application which entered the national stage after compliance with 35 U.S.C. 371 from an international application filed under 35 U.S.C. 363 before November 29, 2000.

(h) If the later-filed application is a nonprovisional application, the reference required by this paragraph must be included in an application data sheet (§ 1.76), or the specification must contain or be amended to contain such reference in the first sentence following the title.

(i) The request for a continued prosecution application under § 1.53(c) is the specific reference required by 35 U.S.C. 120 to the prior-filed application. The identification of an application by application number under this section is the identification of every application assigned that application number necessary for a specific reference required by 35 U.S.C. 120 to every such application assigned that application number."

NOTE: If the new application being transmitted is a divisional, continuation or a continuation-in-part of a parent case, or where the parent case is an international Application which designated the U.S., or benefit of a prior provisional application is claimed, then check the following item and complete and attach **ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED**.

- ☐ The new application being transmitted claims the benefit of prior U.S. application(s). Enclosed are **ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED**

3. Papers Enclosed

A. Required for filing date under 37 C.F.R. § 1.53(b) (Regular) or 37 C.F.R. § 1.153 (Design) Application

30 Pages of specification

4 Pages of claims

9 Sheets of drawing

WARNING: DO NOT submit original drawings. A high quality copy of the drawings should be supplied when filing a patent application. The drawings that are submitted to the Office must be on strong, white, smooth, and non-shiny paper and meet the standards according to § 1.84. If corrections to the drawings are necessary, they should be made to the original drawing and a high-quality copy of the corrected original drawing then submitted to the Office. Only one copy is required or desired. For comments on proposed then-new 37 C.F.R. § 1.84, see Notice of March 8, 1988 (1988 O.G. 57-62).

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NOTE: "Identification of drawings. Identifying indicia, if provided, should include the title of the invention, inventor's name and application number or docket number (if any) if an application number has not been assigned to the application. If this information is provided, it must be placed on the front of each sheet and centered within the top margin."

(complete the following, if applicable)

- ☐ The enclosed drawing(s) are photograph(s).

NOTE: 37 C.F.R. 1.84

"(b) Photographs.

"(1) Black and white. Photographs, including photocopies of photographs, are not ordinarily permitted in utility and design patent applications. The Office will accept photographs in utility and design patent applications, however if photographs are the only practicable medium for illustrating the claimed invention. For example, photographs or photomicrographs of: electrophoresis gels, blots (e.g., immunological, western, Southern, and northern), auto radiographs, cell cultures (stained and unstained), histological tissue cross sections (stained and unstained), animals, plants, in vivo imaging, thin layer chromatography plates, crystalline structures, and, in a design patent application, ornamental effects, are acceptable. If the subject matter of the application admits of illustration by a drawing, the examiner may require a drawing in place of the photograph. The photographs must be of sufficient quality so that all details in the photographs are reproducible in the printed patent.

"(2) Color photographs. Color photographs will be accepted in utility and design patent applications if the conditions for accepting color drawings and black and white photographs have been satisfied. See paragraphs (a)(2) and (a)(1) of this section."

- ☐ The enclosed drawing(s) are in color. Three (3) sets of color drawings and a "PETITION TO ACCEPT COLOR DRAWING(S)" are attached 37 C.F.R. §§ 1.84(a)(2) and 1.84(b)

NOTE: 37 C.F.R. 1.84(b)

"(2) Color. On rare occasions, color drawings may be necessary as the only practical medium by which to disclose the subject matter sought to be patented in a utility or design patent application or the subject matter of a statutory invention registration. The color drawings must be of sufficient quality such that all details in the drawings are reproducible in black and white in the printed patent. Color drawings are not permitted in international applications (see PCT Rule 11.13), or in an application, or copy thereof, submitted under the Office electronic filing system. The Office will accept color drawings in utility or design patent applications and statutory invention registrations only after granting a petition filed under this paragraph explaining why the color drawings are necessary. Any such petition must include the following:

(i) The fee set forth in § 1.17(f);

(ii) Three (3) sets of color drawings;

(iii) A black and white photocopy that accurately depicts, to the extent possible, the subject matter shown in the color drawing, and

(iv) An amendment to the specification to insert (unless the specification contains or has been previously amended to contain) the following language as the first paragraph of the brief description of the drawings:

The patent or application file contains at least one drawing executed in color. Copies of this patent or patent application publication with color drawing(s) will be provided by the Office upon request and payment of the necessary fee."

- ☐ formal
☒ informal

B. Other Papers Enclosed

- 4 Pages of declaration and power of attorney
 1 Pages of abstract
 Other

(New Application Transmittal [4-1]—page 4 of 15)

4. Additional papers enclosed

- ☐ Amendment to claims
 - ☐ Cancel in this applications claims _____ before calculating the filing fee (At least one original independent claim must be retained for filing purposes.)
 - ☐ Add the claims shown on the attached amendment. (Claims added have been numbered consecutively following the highest numbered original claims.)
- ☐ Preliminary Amendment
- ☐ Information Disclosure Statement (37 C.F.R. § 1.98)

NOTE: 37 C.F.R. § 1.97 (c) An information disclosure statement shall be considered by the Office if filed by the applicant within any one of the following time periods.

(1) Within three months of the filing date of a national application other than a continued prosecution application under § 1.53(d),

(2) Within three months of the date of entry of the national stage as set forth in § 1.491 in an international application;

(3) Before the mailing of a first Office action on the merits, or

WARNING: In order to ensure consideration of information previously submitted but which has not been considered in the parent application, an applicant must resubmit the information, complying with 37 C.F.R. § 1.97 and 37 C.F.R. § 1.98, in the continuing application filed under 37 C.F.R. § 1.53(d). See § 608B(c), M.P.E.P., 7th Edition, Rev. 1

- ☐ Form PTO-1449 (PTO/SB/08A and 08B)
- ☐ Citations
- ☐ Declaration of Biological Deposit
- ☐ Submission of "Sequence Listing," computer readable copy and/or amendment pertaining thereto for biotechnology invention containing nucleotide and/or amino acid sequence.
- ☐ Authorization of Attorney(s) to Accept and Follow Instructions from Representative
- ☐ Special Comments
- ☐ Other

5. Declaration or oath (including power of attorney)

NOTE: A newly executed declaration is not required in a continuation or divisional application provided that the prior nonprovisional application contained a declaration as required, the application being filed is by all or fewer than all the inventors named in the prior application, there is no new matter in the application being filed, and a copy of the executed declaration filed in the prior application (showing the signature or an indication thereon that it was signed) is submitted. The copy must be accompanied by a statement requesting deletion of the names of person(s) who are not inventors of the application being filed. If the declaration in the prior application was filed under § 1.47 then a copy of that declaration must be filed accompanied by a copy of the decision granting § 1.47 status or, if a nonsigning person under § 1.47 has subsequently joined in a prior application, then a copy of the subsequently executed declaration must be filed. See 37 C.F.R. §§ 1.63(d)(1)-(3).

NOTE: A declaration filed to complete an application must be executed, identify the specification to which it is directed, identify each inventor by full name including family name and at least one given name, without abbreviation together with any other given name or initial, and the residence, post office address and country or citizenship of each inventor, and state whether the inventor is a sole or joint inventor 37 C.F.R. § 1.63(a)(1)-(4).

NOTE: "The inventorship of a nonprovisional application is that inventorship set forth in the oath or declaration as prescribed by § 1.62, except as provided for in § 1.53(d)(4) and § 1.53(d). If an oath or declaration as prescribed by § 1.63 is not filed during the pendency of a nonprovisional application, the inventorship is that inventorship set forth in the application papers filed pursuant to § 1.53(d), unless a petition under this paragraph accompanied by the fee set forth in § 1.17(f) is filed supplying or changing the name or names of the inventor or inventors." 37 C.F.R. § 1.41(a)(1).

(New Application Transmittal [4-1]—page 6 of 16)

- ☒ Enclosed
Executed by

(check all applicable boxes)

- ☒ Inventor(s) (Ramani Narayan)
☐ legal representative of inventor(s). 37 C.F.R. §§ 1.42 or 1.43.
☐ joint inventor or person showing a proprietary interest on behalf of inventor who refused to sign or cannot be reached
☐ This is the petition required by 37 C.F.R. § 1.47 and the statement required by 37 C.F.R. § 1.47 is also attached. See item 13 below for fee
☐ Not Enclosed.

NOTE: Where the filing is a continuation in the U.S. of an International Application or where the completion of the U.S. application contains subject matter in addition to the International Application, the application may be treated as a continuation or continuation-in-part, as the case may be, utilizing **ADDED PAGE FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION CLAIMED.**

- ☐ Application is made by a person authorized under 37 C.F.R. § 1.41(c) on behalf of all the above named inventor(s)

(The declaration or oath, along with the surcharge required by 37 C.F.R. § 1.16(e) can be filed subsequently).

- ☐ Showing that the filing is authorized
(not required unless called into question. 37 C.F.R. § 1.41(d))

6. Inventorship Statement

WARNING: If the named inventors are each not the inventors of all the claims an explanation, including the ownership of the various claims at the time the last claimed invention was made, should be submitted.

The inventorship for all the claims in this application are:

- ☒ The same.

or

- ☐ Not the same. An explanation, including the ownership of the various claims at the time the last claimed invention was made,
☐ is submitted.
☐ will be submitted

7. Language

NOTE: An application including a signed oath or declaration may be filed in a language other than English. An English translation of the non-English language application and the processing fee of \$130.00 required by 37 C.F.R. § 1.17(h) is required to be filed with the application, or within such time as may be set by the Office. 37 C.F.R. § 1.52(f).

- ☒ English
☐ Non-English
☐ The attached translation includes a statement that the translation is accurate. 37 C.F.R. § 1.52(d).

(New Application Transmitted [4-1]—page 6 of 15)

8. Assignment

Board of Trustees of
MICHIGAN STATE UNIVERSITY

☒ An assignment of the invention to 238 Administration Building, East Lansing, MI 48824-1046

☐ is attached. A separate ☐ "COVER SHEET FOR ASSIGNMENT (DOCUMENT) ACCOMPANYING NEW PATENT APPLICATION" or ☐ FORM PTO 1585 is also attached

☒ will follow

NOTE: "If an assignment is submitted with a new application, send two separate letters—one for the application and one for the assignment." Notice of May 4, 1990 (1114 O.G. 77-78).

WARNING: A newly executed "CERTIFICATE UNDER 37 C.F.R. § 3.73(b)" must be filed when a continuation-in-part application is filed by an assignee. Notice of April 30, 1993, 1180 O.G. 62-64.

☐ This is a ☐ continuation ☐ divisional application and the assignment document for the parent application 0 / _____ was filed on _____

Reel _____

Frame _____

9. Certified Copy

Certified copy(ies) of application(s)

Country	Appl. No.	Filed
Country	Appl. No.	Filed
Country	Appl. No.	Filed

from which priority is claimed

☐ is (are) attached

☐ will follow

NOTE: 37 C.F.R. § 1.55 Claim for foreign priority.

"(b) ...

(1)(i) In an original application filed under 35 U.S.C. 111(a), the claim for priority must be presented during the pendency of the application, and within the later of four months from the actual filing date of the application or sixteen months from the filing date of the prior foreign application. This time period is not extendable. The claim must identify the foreign application for which priority is claimed, as well as any foreign application for the same subject matter and having a filing date before that of the application for which priority is claimed, by specifying the application number country (or intellectual property authority), day, month, and year of its filing. The time periods in this paragraph do not apply in an application under 35 U.S.C. 111(a) if the application is:

(A) A design application, or

(B) An application filed before November 29, 2000.

...

(c) Unless such claim is accepted in accordance with the provisions of this paragraph, any claim for priority under 35 U.S.C. 119(a)-(d) or 365(a) not presented within the time period provided by paragraph (b) of this section is considered to have been waived. If a claim for priority under 35 U.S.C. 119(a)-(d) or 365(a) is presented after the time period provided by paragraph (b) of this section, the claim may be accepted if the claim identifying the prior foreign application by specifying its application number country (or intellectual property authority), and the day, month, and year of its filing was unintentionally delayed. A petition to accept a delayed claim for priority under 35 U.S.C. 119(a)-(d) or 365(a) must be accompanied by:

(New Application Transmittal [4-1]—page 7 of 16)

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(1) The claim under 35 U.S.C. 118(a)-(d) or 365(a) and this section to the prior foreign application, unless previously submitted;

(2) The surcharge set forth in § 1 178; and

(3) A statement that the entire delay between the date the claim was due under paragraph (a)(1) of this section and the date the claim was filed was unintentional. The Commissioner may require additional information where there is a question whether the delay was unintentional."

NOTE: 37 C.F.R. § 1.63 Oath or declaration.

"(a) An oath or declaration filed under § 1 51(b)(2) as a part of a nonprovisional application must:

(c) Unless such information is supplied on an application data sheet in accordance with § 1 78, the oath or declaration must also identify:

(2) Any foreign application for patent (or inventor's certificate) for which a claim for priority is made pursuant to § 1 85, and any foreign application having a filing date before that of the application on which priority is claimed, by specifying the application number country, day, month, and year of its filing."

The foreign application forming the basis for the claim for priority must be referred to in the oath or declaration. 37 C.F.R. § 1.65(a) and 1 63.

NOTE: This item is for any foreign priority for which the application being filed directly relates. If any parent U.S. application or International Application from which this application claims benefit under 35 U.S.C. § 120 is itself entitled to priority from a prior foreign application, then complete item 18 on the ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED.

10. Fee Calculation

NOTE: If the filing fee (or national fee) is paid on filing, but the search fee and/or examination fee is missing, the USPTO will issue a notice requiring that any missing search fee and examination fee (but no surcharge until further notice) be paid within a specified period of time in order to avoid abandonment. Thus, if at least the full basic filing fee under the Consolidated Appropriations Act is paid on or after December 8, 2004, the USPTO will issue a notice requiring any balance of the search fee and the examination fee (but no surcharge).

A. Utility Application

☒ Basic, Examination, Search and Additional Page Fee

☒ Basic fee (\$ 300.00) \$ 300.00

WARNING: The USPTO is considering changing the amount of the search fee and examination fee charged in national stage in the near future. Please refer to www.uspto.gov for the current fees.

☒ Examination fee (\$200.00) \$ 200.00

☒ Fees for Claims Search Fee \$ 500.00

CLAIMS AS FILED					
Claims	Number filed		Number Extra	Rate	
Total Claims	26 --20=	6	x	\$ 60.00	\$ 300.00
Independent Claims	2 -- 3 =	0	x	\$ 200.00	\$ -0-
	Multiple DEPENDENT CLAIMS if applicable	0	+	\$360.00	\$ -0-

☐ Additional fee for specification and drawings filed in paper over 100 sheets (excluding sequence listing or computer program listing filed in an electronic medium) The fee is \$250 for each additional 50 sheets of paper or fraction thereof

U2

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Total Sheets	Extra Sheets	Number of each additional 50 or fraction thereof (rounded up to a whole number)	Rate
-	100	/50	+\$ 250.00
			\$ _____

- ☐ Amendment cancelling extra claims is enclosed
- ☐ Amendment deleting multiple-dependencies is enclosed.
- ☐ Fee for extra claims is not being paid at this time.

NOTE: If the fees for extra claims are not paid on filing they must be paid or the claims cancelled by amendment, prior to the expiration of the time period set for response by the Patent and Trademark Office in any notice of fee delinquency. 37 C.F.R. § 1.164.

Total utility filing fee calculation \$ 1,300

- B. ☐ Design application
(\$200.00—37 C.F.R. § 1.16(f))
- ☐ Design Search Fee (\$100.00) \$ _____
- ☐ Design Examination Fee (\$130.00) \$ _____

Total design fee calculation \$ _____

- C. ☐ Plant application
(\$200.00—37 C.F.R. § 1.16(g))
- ☐ Plant Search Fee (\$300.00) \$ _____
- ☐ Plant Examination Fee (\$180.00) \$ _____

Total plant fee calculation \$ _____

11. Assertion of Small Entity Status

- ☒ Applicant hereby asserts status as a small entity under 37 C.F.R. § 1.27

NOTE: 37 C.F.R. § 1.27(c) deals with the assertion of small entity status, whether by a written specific declaration thereof or by payment as a small entity of the basic filing fee or the fee for the entry into the national phase and states:

"(c) Assertion of small entity status. Any party (person, small business concern or nonprofit organization) should make a determination, pursuant to paragraph (f) of this section, of entitlement to be accorded small entity status based on the definitions set forth in paragraph (e) of this section, and must, in order to establish small entity status for the purpose of paying small entity fees, actually make an assertion of entitlement to small entity status, in the manner set forth in paragraphs (c)(1) or (c)(2) of this section, in the application or patent in which such small entity fees are to be paid.

(1) Assertion by writing. Small entity status may be established by a written assertion of entitlement to small entity status. A written assertion must:

(A) Be clearly identifiable;

(B) Be signed (see paragraph (c)(2) of this section); and

(C) Convey the concept of entitlement to small entity status, such as by stating that applicant is a small entity, or that small entity status is entitled to be asserted for the application or patent. While no specific words or wording are required to assert small entity status, the intent to assert small entity status must be clearly indicated in order to comply with the assertion requirement.

(2) Parties who can sign and file the written assertion. The written assertion can be signed by:

(A) One of the parties identified in § 1.33(b) (e.g., an attorney or agent registered with the Office), § 3.73(b) of this chapter notwithstanding, who can also file the written assertion,

(B) At least one of the individuals identified as an inventor (even though a § 1.63 executed oath or declaration has not been submitted), notwithstanding § 1.33(b)(4), who can also file the written assertion pursuant to the exception under § 1.33(b) of this part; or

(C) An assignee of an undivided part interest, notwithstanding §§ 1.33(b)(3) and 3.73(b) of this chapter, but the partial assignee cannot file the assertion without resort to a party identified under § 1.33(b) of this part.

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(3) Assertion by payment of the small entity basic filing or basic national fee. The payment, by any party, of the exact amount of one of the small entity basic filing fees set forth in §§ 1.18(a), (b), (c), (f), or (g), or one of the small entity basic national fees set forth in §§ 1.402(a)(1), (a)(2), (a)(3), (a)(4), or (a)(5), will be treated as a written assertion of entitlement to small entity status even if the type of basic filing or basic national fee is inadvertently selected in error.

(i) If the Office accords small entity status based on payment of a small entity basic filing or basic national fee under paragraph (c)(3) of this section that is not applicable to that application, any balance of the small entity fee that is applicable to that application will be due along with the appropriate surcharge set forth in § 1.18(h), or § 1.18(i).

(j) The payment of any small entity fee other than those set forth in paragraph (c)(3) of this section (whether in the exact fee amount or not) will not be treated as a written assertion of entitlement to small entity status and will not be sufficient to establish small entity status in an application or a patent."

WARNING: 37 C.F.R. § 1.27(c)(4): "Assertion required in related, continuing, and reissue applications. Status as a small entity must be specifically established by an assertion in each related, continuing and reissue application in which status is appropriate and desired. Status as a small entity in one application or patent does not affect the status of any other application or patent, regardless of the relationship of the applications or patents. The refiling of an application under § 1.53 as a continuation, divisional, or continuation-in-part application (including a continued prosecution application under § 1.53(a)), or the filing of a reissue application, requires a new assertion as to continued entitlement to small entity status for the continuing or reissue application."

WARNING: "Small entity status must not be established when the person or persons signing the statement can unequivocally make the required self-certification." M.P.E.P., § 808.03 (emphasis added).

(complete the following, if applicable)

- ☐ Status as a small entity was asserted in the prior application

_____ / _____, filed on _____, from which benefit is being claimed for this application under

- 35 U.S.C. § ☐ 119(e)
☐ 120
☐ 121
☐ 365(c)

and which status as a small entity is still proper and asserted for this application

- ☐ A copy of the written assertion of small entity filed in the prior application is included

NOTE: A refund based on establishment of small entity status, of a portion of fees timely paid in full prior to establishing status as a small entity may only be obtained if an assertion under § 1.27(c) and a request for a refund of the excess amount are filed within three months of the date of the timely payment of the full fee. The three-month time period is not extendable under § 1.136. 37 C.F.R. § 1.204.

Filing Fee Calculation (50% of A, B or C above)

\$ 650.00

12. Request for International-Type Search (37 C.F.R. § 1.104(d))

(complete, if applicable)

- ☐ Please prepare an international-type search report for this application at the time when national examination on the merits takes place.

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13. Fee Payment Made at This Time

NOTE: If the basic fee is not paid, then a surcharge is required for a subsequent payment as required by 37 C.F.R. § 1.18(h).

- ☒ 37 C.F.R. § 1.18 (a), (b), or (c) (basic fee) \$ 150.00
- ☒ 37 C.F.R. § 1.18 (h), (i), or (j) (presentation of extra claims). 150 00 \$
- ☒ 37 C.F.R. § 1.18 (k), (l), or (m) (search fee) \$ 250.00
- ☒ 37 C.F.R. § 1.18(o), (p), or (q) (examination fee) \$ 100.00
- ☐ 37 C.F.R. 1.18(s) (additional fee for specification and drawings filed in paper over 100 sheets) \$
- ☐ Recording assignment
(\$40.00; 37 C.F.R. § 1.21(h))
(See attached "COVER SHEET FOR
ASSIGNMENT ACCOMPANYING NEW
APPLICATION".) \$
- ☐ Petition fee for filing by other than all the
inventors or person on behalf of the inventor
where inventor refused to sign or cannot be
reached
(\$200.00; 37 C.F.R. §§ 1.47 and 1.17(g)) \$
- ☐ For processing an application with a
specification in
a non-English language
(\$130.00; 37 C.F.R. §§ 1.52(d) and 1.17(i)) \$
- ☐ Processing and retention fee — abandoned
application
(\$130.00; 37 C.F.R. §§ 1.53(d) and 1.21(i)) \$
- ☐ Fee for international-type search report
(\$40.00; 37 C.F.R. § 1.21(e)) \$

NOTE: 37 C.F.R. § 1.21(f) establishes a fee for processing and retaining any application that is abandoned for failing to complete the application pursuant to 37 C.F.R. § 1.53(f) and this, as well as the changes to 37 C.F.R. §§ 1.53 and 1.78(j)(1), indicate that in order to obtain the benefit of a prior U.S. application, either the basic filing fee must be paid, or the processing and retention fee of § 1.21(f) must be paid, within 1 year from notification under § 53(f).

NOTE: If the basic fee is not paid, then a surcharge is required for a subsequent payment as required by 37 C.F.R. § 1.18(h).

Total fees enclosed \$ 650.00

14. Method of Payment of Fees

- ☒ Attached is a ☒ check ☐ money order in the amount of \$ 650.00
- ☐ Authorization is hereby made to charge the amount of \$
- ☐ to Deposit Account No

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- ☐ to Credit card as shown on the attached credit card information authorization form PTO-2038.

WARNING: Credit card information should not be included on this form as it may become public.

- ☐ Charge any additional fees required by this paper or credit any overpayment in the manner authorized above.

A duplicate of this paper is attached

18. Authorization to Charge Additional Fees

WARNING: If no fees are to be paid on filing, the following items should not be completed.

WARNING: Accurately count claims, especially multiple dependent claims, to avoid unexpected high charges, if extra claim charges are authorized.

WARNING: Even though small entity status is accorded where the wrong type of small entity basic filing fee or basic national fee is selected but the exact amount of the fee is paid, applicant still needs to pay the correct small entity amount for the basic filing or basic national fee where selection of the wrong type of fee results in a deficiency. While an accompanying general authorization to charge any additional fees suffices to pay the balance due of the proper small entity basic filing or basic national fee, specific authorizations to charge fees under § 1.17 or extension of time fees do not suffice to pay any balance due of the proper small entity basic filing or basic national fee because they do not actually authorize payment of small entity amounts. *Changes To Implement the Patent Business Goals; Final Rule (Fed. Reg., September 8, 2000, pages 64803-64883, at 64811, O.G. October 3, 2000, pages 14-38).*

- ☒ The Office is hereby authorized to charge, in the manner shown above, the following additional fees that may be required by this paper and during the entire pendency of this application

- ☒ 37 C.F.R. § 1.16 (a), (b), (c) (basic fee).
☒ 37 C.F.R. § 1.16 (h), (i), (j) (presentation of extra claims).
☒ 37 C.F.R. § 1.16 (k), (l), or (m) (search fee)
☒ 37 C.F.R. § 1.16(c), (p) or (q) (examination fee)

NOTE: Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must only be paid or these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 C.F.R. § 1.16(d)), it might be best not to authorize the PTO to charge additional claim fees, except possibly when dealing with amendments after final action.

- ☐ 37 C.F.R. 1.16(a) (additional fee for specification and drawings filed in paper over 100 sheets).
☒ 37 C.F.R. § 1.16(e) (surcharge for filing the basic fee and/or declaration on a date later than the filing date of the application)
☒ 37 C.F.R. § 1.17(a)(1)-(5) (extension fees pursuant to § 1.136(a)).
☒ 37 C.F.R. § 1.17 (application processing fees)

NOTE: "A written request may be submitted in an application that is an authorization to treat any concurrent or future reply, requiring a petition for an extension of time under this paragraph for its timely submission, as incorporating a petition for extension of time for the appropriate length of time. An authorization to charge all required fees, fees under § 1.17 or all required extension of time fees will be treated as a constructive petition for an extension of time in any concurrent or future reply requiring a petition for an extension of time under this paragraph for its timely submission. Submission of the fee set forth in § 1.17(h) will also be treated as a constructive petition for an extension of time in any concurrent reply requiring a petition for an extension of time under this paragraph for its timely submission." 37 C.F.R. § 1.136(h)(3).

- ☐ 37 C.F.R. § 1.18 (issue fee at or before mailing of Notice of Allowance, pursuant to 37 C.F.R. § 1.311(b))

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NOTE: Section 1.311(f) provides that an authorization to charge the issue fee (§ 1.15) to a deposit account may be filed in an individual application only after the mailing of the notice of allowance. Accordingly, general authorizations to pay fees and specific authorizations to pay the issue fee that are filed prior to the mailing of a notice of allowance will generally not be treated as requesting payment of the issue fee and will not be given effect to act as a reply to the notice of allowance. Applicant, when paying the issue fee, should submit a new authorization to charge fees, such as by completing box 8b on the current PTOL-85B form. Where no reply to the notice of allowance is received, the application will stand abandoned notwithstanding the presence of general authorizations to pay fees or a specific authorization to pay the issue fee that were submitted prior to mailing of the notice of allowance. Where an attempt is made to pay the issue fee but an incorrect amount is submitted, § 1.311(f)(1), or where the Office's issue fee transmittal form (currently PTOL-85(B)) is completed by applicant and submitted, § 1.311(f)(2), in reply to a notice of allowance, an exception will be made. Such submissions will operate as a request to charge the issue fee to any deposit account identified in a previously filed (i.e., submitted prior to the mailing of the notice of allowance) authorization to charge fees, and will be allowed to act as payment of the correct issue fee. § 1.311(f). See also the change to § 1.28(f). Notice of September 8, 2000, Fed. Reg. 54803-54803, at 54846 and 54847

NOTE: 37 C.F.R. § 1.28(b) requires "Notification of any change in status resulting in loss of entitlement to small entity status must be filed in the application prior to paying, or at the time of paying, the issue fee." From the wording of 37 C.F.R. § 1.28(b), (a) notification of change of status must be made even if the fee is paid as "other than a small entity" and (b) no notification is required if the change is to another small entity.

18. Instructions as to Overpayment

NOTE: "Amounts of twenty-five dollars or less will not be returned unless specifically requested within a reasonable time, nor will the payer be notified of such amounts, amounts over twenty-five dollars may be returned by check or, if requested, by credit to a deposit account." 37 C.F.R. § 1.28(f).

- ☒ Credit Account No. 13-0610
☐ Refund

Reg No. 20,931

Tel No (517) 347-4100

Customer No. 21036



SIGNATURE OF PRACTITIONER
Ian C. McLeod

(type or print name of attorney)
2190 Commons Parkway

P.O. Address
Okemos, Michigan 48864

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☐ **Incorporation by reference of added pages**

(check the following item if the application in this transmittal claims the benefit of prior U.S. application(s) (including an international application entering the U.S. stage as a continuation, divisional or C-I-P application) and complete and attach the ADDED PAGES FOR NEW APPLICATION TRANSMITTAL WHERE BENEFIT OF PRIOR U.S. APPLICATION(S) CLAIMED)

- ☐ **Plus Added Pages for New Application Transmittal Where Benefit of Prior U.S. Application(s) Claimed**

Number of pages added _____

- ☐ **Plus Added Pages for Papers Referred to in Item 4 Above**

Number of pages added _____

- ☐ **Plus added pages deleting names of inventor(s) named in prior application(s) who is/are no longer inventor(s) of the subject matter claimed in this application.**

Number of pages added _____

- ☐ **Plus "Assignment Cover Letter Accompanying New Application"**

Number of pages added _____

☒ **Statement Where No Further Pages Added**

(if no further pages form a part of this Transmittal, then end this Transmittal with this page and check the following item)

- ☒ **This transmittal ends with this page**

**ADDED PAGE(S) FOR SPECIAL COMMENTS FOR NEW APPLICATION
TRANSMITTAL**

Added page _____

(Added Page(s) for Special Comments for New Application Transmittal [4-1]—page 15 of 16)

**STARCH-VEGETABLE OIL GRAFT
COPOLYMERS AND THEIR BIOFIBER COMPOSITES, AND A
PROCESS FOR THEIR MANUFACTURE**

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] Not Applicable

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

[0002] Not Applicable

STATEMENT REGARDING GOVERNMENT RIGHTS

[0003] Not Applicable

BACKGROUND OF THE INVENTION

(1) Field of the Invention

[0004] This invention relates to the preparation of thermoplastic starch-vegetable oil graft copolymers and moldable compositions containing a biofiber reinforced in the starch-vegetable oil graft copolymer matrix, which are preferably biodegradable. More specifically, the present invention relates to a process of making biofiber reinforced starch-vegetable oil graft copolymer blends which can be injection molded to give products which are totally biodegradable, easily processed and possessing good mechanical properties.

(2) Description of Related Art

[0005] Traditionally, plastics have been formulated to result in strong, light-weight, durable, and bioresistant polymeric materials. It is the durability and indestructibility that makes plastic the material of choice for many applications. However, these same properties are problems when

the plastics enter the waste stream. The recent trend is to create biodegradable plastics, most of such plastics being first commercialized in the mid 1980's (Barenberg, S A, et al, "Degradable Materials Perspectives, Issues and Opportunities" (1990 and Vert, M, et al, "Biodegradable Polymers and Plastics" (1992))

[0006] Among the first 'biodegradable' plastics made were blends of non-biodegradable polyolefins with starch which were at best only partially biodegradable (Gilmore, D F, et al, "The Fate of 'Biodegradable' Plastics in municipal Leaf Compost", J Industr Microbiol 10, 199-206 (1992), Barak, Y, et al, J Environ Qual, 20 173 (1991), Krupp, L R, et al, Environ Sci Technol 26 193 (1992), and Swanson, C L, et al, J Environm Polymer Degrad 1(2), 155-165 (1993). These plastics are not compatible with waste management infrastructures, such as composting. Moreover, at that time, the appropriate infrastructures capable of dealing with biodegradables did not exist. Instead of composting, these products generally ended up in sanitary landfills.

[0007] Landfills, in general, are a poor choice as a repository of plastic and organic waste. Landfills are plastic-lined tombs designed to retard biodegradation by providing little or no moisture with negligible microbial activity. Organic waste, such as lawn and yard waste, paper, and food waste should not be entombed in such landfills to be preserved for posterity. Accordingly, there is a growing trend to divert these materials into composting facilities, which allow them to be biodegraded to produce humus or compost. This compost can then be used as a valuable soil additive for new plant growth.

[0008] When plastics are designed to be biodegradable, utilizing renewable resources as the major raw material component, the plastics can become part of an ecologically

sound solution

[0009] Biodegradation of natural materials produces valuable compost as the major product, in addition to water and carbon dioxide. Such carbon dioxide is fixed or neutral and therefore does not contribute to an increase in the greenhouse gases.

[0010] U S Patent No 5,095,054 to Lay et al (1992) discloses the use of water as a plasticizer for starch (referred to as starch "destructurization") in order to make the material processable in for example an extruder. Products derived therefrom tend to have the problem of rapidly losing water to the environment by evaporation. As a result, this type of material tends to become brittle with age. These materials are also highly water sensitive which is undesirable in the majority of applications of thermoplastic products. To address this issue of water sensitivity, the patent also includes various blends of destructured starch with a variety of synthetic petroleum-based plastics. Such blend compositions, along with earlier starch-filled polyolefins, are at best only biodegradable and not fully biodegradable. Starch-polyolefin compositions have been reported by the Fertec group (PCT Int Pat Appl WO 92/14782, Bastioli, et al "Biodegradable Compositions Based on Starch and Thermoplastic Polymers", Novamont S P A, (1992), PCT Int Pat Appl WO 91/02025, C Bastioli, et al, "A Polymer Composition including Destructured Starch and an Ethylene Copolymer", Novamont S P A (1991) (See also WO 91/02023 and WO 91/02024)).

[0011] U S Patent No 4,873,270 to Aime et al, (1989), describes blends of polyurethane with for example poly(vinylchloride) and a carbohydrate such as potato flour. U S Patent Nos 3,850,862 and 3,850,863 to Clendinning et al (1974), disclose blends of a naturally biodegradable product, such as tree bark, protein, starch, peat moss, saw dust, etc, with a dialkanoyl polymer (Patent No 3,850,862) such as

poly(alkylene adipate), or with an oxyalkanoyl polymer (Patent No 3,850,863), such as poly(caprolactone) U S Patent No 4,863,655 to Lacourse et al (1989) discloses water-soluble high amylose starch containing poly(vinyl alcohol) This type of modified starch is highly hydrophilic and water soluble, the starch contains about 5% by weight propylene oxide corresponding to a theoretical degree of substitution of 0.19 This is a very low degree of substitution compared with the maximum degree of substitution for starch which is 3.0 according to the three available hydroxyl groups on the anhydroglucose repeat unit The poly(vinyl alcohol) typically used as a blend component further adds to the water-sensitive nature of these materials

[0012] As discussed earlier, prior art blend compositions are at best biodegradable and not fully biodegradable In composting, the non-biodegradable components will be persistent resulting in an irreversible build-up of these components in the environment causing reduced productivity and fertility of the soil Even if such 'biodegradable' blend compositions are partially biodegradable, the resulting compost will have very little value In fact, these recalcitrant components will be present in the final compost at significantly higher concentration levels than in the original waste mixture

[0013] Graft copolymerization of a vinyl monomer on the starch backbone was used to modify starch Fanta and Bagley (Fanta, G F , et al , Encyclopedia of Polymer Science, John Wiley & Sons, New York (1970) and Fanta, G F , Block and Graft Copolymers- Vol 1, John Wiley & Sons, New York (1973) have reviewed the synthesis and discussed some applications of starch graft copolymers Otey et al (Otey, F H , et al , Industrial Engineering Chemistry Products Research Development, 19, 592 (1980), Otey, F H , et al , Industrial Engineering Chemistry Products Research Development, 23, 284 (1984), and

Otey, R P , et al , Industrial Engineering Chemistry Products Research Development, 26, 1659 (1987)) blended starch with poly(ethylene-co-acrylic acid)(EAA) In these papers, the authors suggested the formation of hydrogen bonds between the carboxylic group in EAA and the hydroxyl group in starch Increasing the level of starch decreased the percentage elongation of the film and increased the diffusion rate of water Similar complexes like EAA can also be formed with the hydroxyl groups of the polyethylene-vinyl alcohol (EVOH) copolymer Bloembergen et al have reported on blends and alloys containing lignocelluloses like starch, cellulose acetate etc U S Patent No 5,314,934 to Tomka et al provides a process to produce a polymer mixture essentially consisting of thermoplastically processable starch (up to 70-wt%) During this mixture process, an esterification reaction takes place between the maleic anhydride groups in the terpolymer with free hydroxyl groups in the starch These reactive blends can be blown into film with properties comparable to LDPE U S Patent No 5,234,977 to Bastioli et al discloses a material used for the product of biodegradable articles in film, sheet or fiber form, which can be produced by extrusion from a molten mass that includes a synthetic thermoplastic polymer and a destructured starch to which a boron containing compound such as boric acid has been added

[0014] U S Patent Nos 5,618,341, 5,679,145, 5,858,824, 6,168,837 and 6,231,970 to Andersen et al explain thermoplastic starch compositions that include a particulate filler and an optional fiber These compositions may be shaped into a wide variety of useful articles, such as sheets, films, containers and packaging materials The overall composition in this case is typically more environmentally friendly compared to conventional thermoplastic materials

[0015] U S Patent No 5,928,737 to Hammer et al discloses

thermoplastic starch compositions formed by heating granular starch with an additive and further reinforced with fiber and protein to form sausage casings

[0016] U S Patent No 6,521,147 to Arentsen et al provides a method and apparatus for manufacturing fiber-reinforced foamed paper-like products Further, U S Patent No 6,565,640 to Bengs et al provides compositions based on starch and/or modified starch and comprising plasticizers which can be processed thermoplastically to give shaped articles which are biodegradable and physiologically non-hazardous These articles may be used as packaging or casing for food or drink or pharmaceutical products, or also for the controlled release of active substances, or else for producing temporary protective coatings

[0017] More recently, U S Patent No 6,709,526 to Bailey et al provides compositions containing starch, a polymer that is substantially compatible with starch and has a weight-average molecular weight of at least 500,000 g/mol such that the polymer forms effective entanglements or associations with neighboring starch molecules, and preferable at least one additive to improve melt flow and melt processability The composition is especially suitable for uniaxial and biaxial extensional processes to make fibers, films, foams and like products

[0018] The prior art on biodegradable materials and blends is restricted to starch-based materials in which the starch component is hydrophilic (water sensitive) No prior art exists on blends containing starch oil graft copolymer products and their biofiber reinforced composites as fully biodegradable products which are readily processable on conventional plastics processing equipment such as extruders injection molders, etc

OBJECTS

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[0019] It is an object of the present invention to achieve good processability and mechanical properties with a novel starch graft co-polymer product. It is further an object to provide such processable products which are also biodegradable. It is also an object to provide new starch-based materials which utilize agricultural resources and return those resources to nature in an environmentally sound manner. These and other objects will become increasingly apparent by reference to the following description. The substance and advantages of the present invention will become increasingly apparent by reference to the following description and the drawings.

SUMMARY OF THE INVENTION

[0020] The present invention provides new biobased polymeric materials which are sustainable and environmentally responsible. The present invention describes a method of preparing a blend system which is compatibilized and contains fully biodegradable components for making moldable products. The compatibilized blend system has been designed and engineered by blending biodegradable, starch-vegetable oil graft copolymers with biofibers and optionally biodegradable aliphatic and aliphatic-aromatic copolymers of polyesters. Examples of biodegradable polyesters include poly(caprolactone) (PCL), poly(vinylacetate-co-vinylalcohol) (PVAc/VA), poly(lactic acid) or polylactide (PLA), poly(glycolic acid) or polyglycolide (PGA), and related like polybutylene adipate coterphthalate (PBAT), copolyesters including the various combinations of stereoisomers, bacterial and synthetic poly(beta-hydroxybutyrate) (PHB), Poly(beta-hydroxybutyrate-co-beta-hydroxyvalerate (PHB/V), and other poly(beta-hydroxyalkanotes) (PHA).

[0021] The present invention provides a preferred method for obtaining starch-vegetable oil graft copolymer formed by

reacting starch with a vegetable oil in the presence of a plasticizer using free-radical initiator and further reinforcing it with a fiber and optionally biodegradable polyester. The plasticizer is selected from the group including a variety of biodegradable esters miscible with the starch ester and polyester. These products are readily processable, and have good mechanical properties. Most importantly, the polymers are fully biodegradable and after their end-use can be composted to produce valuable soil amendment.

[0022] The present invention relates to a biodegradable starch-vegetable oil graft copolymer composition which is a reactively blended mixture comprising a granular starch, a vegetable oil or a derivative thereof comprising a modifier reactive with the starch, and a plasticizer. The present invention relates to the composition wherein the granular starch is selected from the group consisting of cornstarch, potato starch, tapioca starch, rice starch, wheat starch, cassava starch and high amylase cornstarch. Preferably the starch in the mixture is 5% to 90% by weight. Preferably the starch has a moisture content of about 0.5% to 15% by weight of the starch. Preferably the starch is in a pulverized form. Preferably the starch is chemically modified with the modifier. Generally the vegetable oil is unsaturated and is selected from the group consisting of fatty acids and esters. The derivatives of vegetable oil are generally reactive in the composition. Epoxylated, maleated and hydroxylated vegetable oils are the most preferred. Preferably the vegetable oil is selected from the group consisting of corn oil, soybean oil, maleated soybean oil and epoxidized soybean oil and mixtures thereof. Preferably the vegetable oil is 1% to 10% by weight of the composition. Preferably the plasticizer is a polyhydric alcohol in an amount between 10 to 40 wt-% of the total of the composition.

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Preferably a free-radical peroxide initiator is provided in the mixture, and wherein the free-radical initiator is 2,5-Bis(tert-butylperoxy)-2,5-dimethylhexane (LUPEROX 101) Preferably a free radical peroxide initiator is provided in the mixture in an amount between about 0.01 to 3-wt% of the total weight of the composition. Preferably the composition comprises a fiber, and wherein the fiber is selected from the group consisting of cotton, wood, flax, abaca, sisal, ramie, hemp, and bagasse. Preferably the fiber has an aspect ratio of at least about 10:1. Preferably the fiber is present in an amount between about 3% to 80% by weight of the composition. Preferably the composition comprising the modifier which is selected from the group consisting of cyclic or dibasic acid and anhydrides thereof. Preferably the modifier is present in the composition in amount of about 0.1 to about 10% by weight of the starch. Preferably the composition comprises a biodegradable polyester selected from the group consisting of poly(caprolactone) (PCL), poly(vinylacetate-co-vinylalcohol) (PVAc/VA), poly(lactic acid) or polylactide (PLA), poly(glycolic acid) or polyglycolide (PGA), poly(beta-hydroxyalkanoates) (PHA), and aliphatic aromatic copolyesters, like polybutylene adipate coterphthalate (PBAT). Preferably the biodegradable polyester is 1% to 60% by weight of the composition.

[0023] The present invention further relates to a process for producing a biodegradable starch vegetable oil copolymer composition which comprises reactively blending a mixture of a granular starch, a vegetable oil or a derivative thereof comprising a modifier reactive with the starch, and a plasticizer in a reactor, and removing the composition from the reactor to produce the composition.

[0024] Preferably the reactor is an extruder. Preferably the vegetable oil derivative is a maleated vegetable oil.

Preferably the mixture further comprises a filler Preferably the filler is a natural fiber Preferably the plasticizer is a solvent for the mixture

BRIEF DESCRIPTION OF THE FIGURES

[0025] Figure 1 illustrates the screw configuration used in the synthesis of starch-vegetable oil graft copolymers by reactive extrusion processing The screw of length 12 5 D is followed by the screw of length 15 5 D and then finally by the screw of length 12 D D is 30 mm

[0026] Figure 2 shows the FTIR scan of extracted product confirming reactivity between starch and corn oil in the synthesis of starch-corn oil graft copolymers FTIR scans for pure starch and corn oil are also shown as a comparison

[0027] Figure 3 shows the FTIR scan of extracted product confirming reactivity between starch and maleated corn oil in the synthesis of starch-corn oil graft copolymers (absence of free-radical initiator)

[0028] Figure 4 shows the ESEM (scanning electron microscope) image of regular corn starch

[0029] Figure 5 shows the ESEM image of starch-maleated corn oil graft copolymer produced by reactive extrusion processing

[0030] Figure 6 depicts the ESEM image of fiber reinforced starch-vegetable oil graft copolymer

[0031] Figure 7 shows the ESEM image of fiber reinforced starch-vegetable oil graft copolymers containing polycaprolactone polyester

[0032] Figure 8 is a bar graph showing the tensile strength properties of various fiber reinforced starch-vegetable oil graft copolymer blends as explained in Examples 5-9

[0033] Figure 9 is a bar graph showing the break elongation properties of various fiber reinforced starch-vegetable oil

graft copolymer blends as explained in Examples 5-9

DESCRIPTION OF PREFERRED EMBODIMENTS

[0034] The present invention provides a biodegradable moldable product wherein, generally, the product is prepared by introducing a reinforcing fiber and an optionally biodegradable polyester into a starch-vegetable oil graft copolymer matrix. Further, the starch-vegetable oil graft copolymer can be prepared by reactive-extrusion processing of starch, vegetable oil, plasticizer and a free-radical initiator.

[0035] The degree of substitution (DS) of a polymer is the (average) number of substituent groups attached per base unit (in the case of condensation polymers) or per monomeric unit (in the case of additional polymers). The term has been used mainly in cellulose chemistry where each anhydroglucose unit has three reactive (hydroxyl) groups. Degree of substitution can then range from zero (cellulose itself) to three (fully substituted cellulose). The same chemistry applies herein to starch wherein each glucose moiety has three reactive hydroxyl sites.

[0036] Starch $(C_6H_{10}O_5)_n$ is a mixture of linear (amylose) and branched (amylopectin) polymers. Amylose is essentially a linear polymer of $\alpha(1-4)$ linked D-glucopyranosyl units. Amylopectin is a highly-branched polymer of D-glucopyranosyl units containing $\alpha(1-4)$ linkages, with $\alpha(1-6)$ linkages at the branch points. The granular starch was obtained from Corn Products, Inc. (Illinois) from the country of Brazil. Various other starches, such as potato starch, tapioca starch, rice starch, wheat starch, cassava starch, and other starches familiar to those skilled in the art can be utilized in accordance with the present invention. Compositions having increased levels of linear starch polymers are most likely to

be the correct choice in terms of processability, molding and mechanical properties

[0037] The starch-vegetable oil graft copolymer compositions of the present invention comprises what is considered in the art to be a specialty starch, preferably having an amylose content greater than 50% and most preferably greater than 70%. The degree of substitution preferably ranges from 0.001 to 1 DS and more preferably from 0.01 to 0.05 DS.

[0038] Modified starches are particularly useful in this invention. By chemically modified, it is meant that the starch can be derivatized or modified by typical processes known in the art, e.g. esterification, etherification, oxidation, acid hydrolysis, crosslinking and enzyme conversion. Typically, modified starches include esters, such as the acetate and half-esters of dicarboxylic acids, particularly the alkylsuccinic acids, ethers, such as the hydroxyethyl and hydroxypropyl starches and cationic starches such as starch modified with 2-diethylaminoethyl chloride (DEC) and starch modified with quaternary ammonium reagents such as 3-chloro-2-hydroxypropyltrimethylammonium chloride, starches oxidized with hypochlorite, starches reacted with crosslinking agents such as phosphorus oxychloride, epichlorohydrin, and phosphate derivatives prepared by reaction with sodium or potassium orthophosphate or tripolyphosphate and combinations thereof. These and other conventional modifications of starch are described in publications such as "Starch Chemistry and Technology", Second Edition, Edited by Roy L. Whistler, et al., Chapter X, Starch Derivations Production and Uses by M. W. Rutenberg, et al., Academic Press, Inc., (1984). The starch component in the blend is 5% to 90% by weight, preferably from 50% to 80% by weight.

[0039] The aforementioned starches and starch esters can be made into a wide range of starch-vegetable oil graft copolymers.

using a wide variety of oils. The oils pertaining to this invention are unsaturated fatty acids (triglycerides) obtained from plant based resources. The preferred oils are corn, soybean and mixtures thereof and their maleated and epoxidized versions. The concentration of the oils in the starch-vegetable oil graft copolymers is 1% to 10% by weight, preferably between 2% to 5% by weight.

[0040] A plasticizer which is a polyhydric alcohol, which is well known to those skilled in the art, can be added to the composition to achieve greater material processability and product flexibility. An obvious requirement of these plasticizers is that they are biodegradable. Optional plasticizers are well known in the art. Typical plasticizers are water, dihydric, trihydric, polyhydric alcohols, and their derivatives. Some examples are ethylene glycol, propylene glycol, glycerol, glycerol esters, erythritol, pentaerythritol, sorbitol, and higher molecular weight plasticizers like poly glycerol and poly glycerol esters. Glycerol esters include glycerol monoacetate, diacetate, and triacetate. For the purpose of this invention, it is preferred that the plasticizers chosen have a boiling point sufficiently higher than the processing temperature in the extruder and be thermally stable. The preferred plasticizers are glycerol, glycerol esters, sorbitol, and mixture thereof. It is preferred that the amount of the plasticizer be in the range of 1 to 30% by weight of the total reactant mixture, preferably 15 to 30% by weight.

[0041] The free-radical initiator used in this invention was obtained from Aldrich Chemical (Wisconsin) and is 2,5-Bis (tert-butylperoxy)-2,5-dimethylhexane (LUPEROX 101, Aldrich, Wisconsin). Numerous other free radical initiators are known to those skilled in the art. The radical initiator is preferably added in an amount ranging between 0.01 to 10-wt% of

the total weight

[0042] It is highly desirable that these starch-vegetable oil graft copolymers be capable of forming biodegradable products with good mechanical properties which can be easily processed in conventional equipments like extruder and injection molder

[0043] These starch-vegetable oil graft copolymers are useful as matrices which can preferably be reinforced with fiber, modifiers and optional biodegradable polyester to form blends suitable to be injection molded

[0044] Preferred fibers include cotton, wood fibers (both hardwood or softwood fibers, examples of which include southern hardwood and southern pine), flax, abaca, sisal, ramie, hemp and bagasse because they readily decompose under normal conditions. The fibers have an aspect ratio of at least about 10:1, preferably greater than about 25:1, more preferably greater than about 100:1, and most preferably greater than about 250:1

[0045] A wide range of fibers can optionally be used in order to improve the physical properties of the thermoplastic starch compositions of the present invention. As used in the specification and the appended claims, the terms "fibers" and "fibrous material" include both inorganic and organic fibers. Fibers may be added to the moldable mixture to increase the toughness, and fracture energy, as well as the flexural and tensile strengths of the resulting sheets and articles. Fibrous materials reduce the likelihood that the sheets, films or articles made from thermoplastic starch compositions will shatter when cross-sectional forces are applied. Accordingly, the concentration of fibers within the thermoplastic starch compositions of the present invention can be included in a broad range from 1% to about 60% by weight of the thermoplastic starch composition. Preferably, fibers will be included in an

amount in a range from about 10% to about 30% by weight of the thermoplastic starch composition

[0046] Reactive modifiers for the composition preferably are selected from the class of cyclic dibasic acid anhydrides such as maleic, succinic, itaconic, phthalic and combinations thereof. Other modifiers include phthalic acid derivatives like diethyl phthalate, dimethyl phthalate, and dioctyl phthalate, phosphoric acid derivatives like triethyl phosphate, tributyl phosphate, and trioctyl phosphate, glycerol, and glycerol esters, and sulfonic acid derivatives. The modifier is preferably present in amount of about 1% to about 10% by weight of the starch based reactant whereby to provide an acidic environment for the mixture.

[0047] Examples of biodegradable polyesters include poly(caprolactone) (PCL), poly(vinylacetate-co-vinylalcohol) (PVAc/VA), poly(lactic acid) or polylactide (PLA), poly(glycolic acid) or polyglycolide (PGA), and related copolyesters including the various combinations of stereoisomers, bacterial and synthetic poly(-hydroxybutyrate) poly(beta-hydroxybutyrate-co-beta-hydroxyvalerate) (PHB/V), and other poly(beta-hydroxyalkanoates) (PHA), and biodegradable aliphatic-aromatic copolyesters. The biodegradable polyester is incorporated in the final blend in an amount ranging from 0% to 60% by weight and preferably from 10% to 30% by weight.

[0048] The products of the present invention possess significant mechanical properties while still retaining their biodegradability. This is further achieved by engineering the appropriate degree of substitution and molecular weight of the starch-vegetable oil graft copolymers component, by blend composition, choice of polyester, modifier and fiber.

[0049] The present invention can be processed by various methods known in the art, such as extrusion pelletizing and injection molding. For example, the biodegradable fiber

reinforced starch-vegetable oil graft copolymers made in accordance with the present invention can be injection molded to give a variety of molded products

[0050] A typical reactive extrusion process to produce starch-vegetable oil graft copolymer in the presence of a plasticizer during free radical initiators is described hereafter 800 gm of regular corn-starch was pre-blended with 20 gm corn oil in a kitchen blender for 15 minutes Meanwhile, 200 gm of glycerol was mixed with 16 gm 2,5-bis(tert-butylperoxy)-2,5-dimethylhexane (LUPEROX 101) in a glass beaker The pre-blended mixture of starch and corn oil was further blended with the glycerol mixture and mixed for around 15 minutes This combined mixture was fed to the feed port of a twin-screw co-rotating CENTURY extruder (screw diameter 30 mm and L/D ratio 40) (Traverse City, MI) Vent port was kept open to remove moisture present A screw configuration as shown in Figure 1 was used for the reactive extrusion processing The processing conditions for the reactive extrusion modification of starch to form starch-vegetable oil graft copolymers are shown in Table 1

TABLE 1

ZONE	SET	ACTUAL	°C
ZONE 1	15	56	°C
ZONE 2	95	99	°C
ZONE 3	125	131	°C
ZONE 4	145	147	°C
ZONE 5	150	157	°C
ZONE 6	155	161	°C
ZONE 7	155	155	°C
ZONE 8	155	147	°C
ZONE 9	150	93	°C
DIE	145	145	°C
MELT TEMP		164	°C
MOTOR SPEED		120	RPM
TORQUE		40-50	%
PRESSURE		40-50	Pisa
FEEDER SPEED, CENTURY			%
FEEDER SPEED, Mini			%
FEEDER SPEED, EXT			%

The extruded strand was collected in aluminum pans and air-cooled before being ground to a fine powder. The starch-vegetable oil graft copolymer was stored in an oven at 50°C to prevent entrainment of moisture.

[0051] A typical reactive extrusion process to produce biofiber reinforced starch-vegetable oil graft copolymer in the presence of optional modifiers such as maleic anhydride is described hereafter. The starch-vegetable oil graft copolymer (pre-blended with 1-wt% maleic anhydride) was blended with Solka floc (Cellulose based fiber) in a 70/30 ratio. The mixture was fed to the feed port of a twin-screw co-rotating CENTURY extruder (screw diameter 30 mm and L/D ratio 40). Vent

port was kept open to remove moisture present. A screw configuration as shown in Figure 1 was used for the reactive extrusion processing. The processing conditions for the process are similar to the one described in Table 1. The extruded strand was air cooled before being palletized in line. The biofiber-reinforced composite is stored in an oven at 75°C to prevent entrainment of moisture.

[0052] Alternatively, the above two process steps can be combined into a one-step operation wherein the starch-vegetable oil graft copolymer is made in the first section of the extruder (LD of 20) followed by the downstream addition of the biofiber to produce biofiber reinforced starch-vegetable oil graft copolymer composites. The composite was injection molded to form test dog bone samples.

[0053] The Injection Molding conditions are shown in Table 2 for the two step process.

TABLE 2

ZONE	SET	ACTUAL	UNITS
ZONE 1	15	38	°C
ZONE 2	95	94	°C
ZONE 3	145	145	°C
ZONE 4	150	148	°C
ZONE 5	155	150	°C
ZONE 6	155	163	°C
ZONE 7	155	158	°C
ZONE 8	155	150	°C
ZONE 9	150	148	°C
DIE	145	146	°C
MELT TEMP		152	°C
MOTOR SPEED		150	RPM
TORQUE		15-20	kg
PRESSURE			Pis
FEEDER SPEED, CENTURY			kg
FEEDER SPEED, MINI			kg
FEEDER SPEED, EXT			kg

[0054] Starch-vegetable oil graft copolymer has been manufactured by the extrusion processing of starch with corn oil in the presence of glycerol as a plasticizer using LUPEROX 101 as a free radical initiator Basic characterizations such as thermal analysis, Fourier Transformed Infra Red Spectroscopy (FTIR) have been completed Biofiber reinforced starch-vegetable oil graft copolymer have been manufactured by reactive extrusion processing as explained above in both a single step as well as a dual step process The biofiber composite has been injection molded to form dog bone test sample bars Mechanical properties have been determined

[0055] The effect of changing parameters such as corn oil

concentration, type of oil, plasticizer type and concentration, temperature and pressure profiles, etc on the reactivity and processing of starch-vegetable oil graft copolymers can easily be determined Further changing the biofiber type or the biofiber addition content and comparing the processing and mechanical properties of the composite with commodity plastics starting from a molding composition is easily within the skill of the art Finally, the reactive extrusion processing of biofiber reinforced starch-vegetable oil graft copolymers can be extended to an industrial scale-up for the manufacture of biodegradable articles for applications such as biodegradable forks, knives, spoons, golf tees and the like

[0056] The following experimental Examples specifically demonstrate the utility of the present invention for forming biodegradable products containing starch, vegetable oil, plasticizer, initiator fiber and biodegradable polyester

[0057] Examples of materials made in accordance with the present invention in compost experiments confirm biodegradability

EXAMPLES

Example 1

[0058] A starch modification reaction was carried out as follows

[0059] 740 gm of granular corn-starch (Corn Products, Inc) was mixed with 71.5 gm of corn oil (Corn Products, Inc) Further, 185.3 gm glycerol was mixed with 3.2 gm of 2,5-Bis (tert-butylperoxy)-2, 5-dimethylhexane (LUPEROX 101 (Aldrich)

The two mixtures were mixed in a blender for 15 minutes and extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40 The temperature profile and the extrusion processing conditions are shown in Table 1 Screw configuration consisting of both conveying and

69 165

kneading elements was used and is shown in Figure 1 $DS = (162*W)/(100*M - (M-1)*W)$, where 162 represents the molecular weight of one anhydroglucose unit, W is the weight % of the substituent and M the molecular weight of the substituent In our case, W=7.18 and M=870 g/mol This translates to a theoretical DS value of 0.014 (i.e. one mol of corn oil per 100 glucose units) The starch-corn oil graft copolymer was extracted for 24 hours in a Soxhlet apparatus using Dichloromethane solvent Corn oil reacted was found to be 60% FTIR scan confirming reactivity is shown in Figure 2

Example 2

[0060] A starch modification reaction following the procedure as explained in Example 1 was carried out as follows

[0061] 740 gm of granular corn-starch (Corn Products, Inc) was mixed with 71.5 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further, 185.3 gm glycerol was mixed with 3.2 gm of 2,5-Bis (tert-butylperoxy)-2,5-dimethylhexane (LUPEROX 101 (Aldrich) The two mixtures were mixed in a blender for 15 minutes and extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40 The temperature profile and the extrusion processing conditions are shown in Table 1 Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1 $DS = (162*W)/(100*M - (M-1)*W)$, where 162 represents the molecular weight of one anhydroglucose unit, W is the weight % of the substituent and M the molecular weight of the substituent In this case, W = 7.18 and M = 968 (870 of CO + 98 of MA) This translates to a theoretical DS value of 0.012 (i.e. one mol of corn oil per 100 glucose units) The starch-corn oil graft copolymer was extracted for 24 hours in a Soxhlet apparatus using Dichloromethane solvent Maleated Corn oil reacted was

found to be 52%

Example 3

[0062] A starch modification reaction following the procedure as explained in Example 1 was carried out as follows

[0063] 740 gm of granular corn-starch (Corn Products, Inc) was mixed with 74.7 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further, 185.3 gm glycerol was added and the mixture was kneaded in a kitchen blender for 15 minutes and extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40. The temperature profile and the extrusion processing conditions are shown in Table 1. Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1. $DS = (162 \cdot W) / (100 \cdot M - (M-1) \cdot W)$, where 162 represents the molecular weight of one anhydroglucose unit, W is the weight % of the substituent and M the molecular weight of the substituent. In this case, $W = 7.18$ and $M = 968$ (870 of CO + 98 of MA). This translates to a theoretical DS value of 0.012 (i.e. one mol of corn oil per 100 glucose units). The starch-corn oil graft copolymer was extracted for 24 hours in a Soxhlet apparatus using Dichloromethane solvent. Maleated Corn oil reacted was found to be 58%. FTIR scan confirmed reactivity even in the absence of Luperox 101 is shown in Figure 3.

Example 4

[0064] A starch modification reaction following the procedure as explained in Example 1 was carried out as follows

[0065] 770 gm of granular corn-starch (Corn Products, Inc) was mixed with 35 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further 185.3 gm glycerol was added and the mixture was kneaded in a kitchen

blender for 15 minutes and extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a LD of 40. The temperature profile and the extrusion processing conditions are shown in Table 1. Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1. $DS = 162 \cdot W / (100 \cdot M - (M-1) \cdot W)$, where 162 represents the molecular weight of one anhydroglucose unit, W is the weight % of the substituent and M the molecular weight of the substituent. In this case, W = 7.18 and M = 968 (870 of CO + 98 of MA). This translates to a theoretical DS value of 0.006 (i.e. one mol of corn oil per 100 glucose units). The starch-corn oil graft copolymer was extracted for 24 hours in a Soxhlet apparatus using Dichloromethane solvent. Maleated Corn oil reacted was found to be 82%. ESEM images of pure starch and starch-vegetable oil graft copolymer are shown in Figures 4 and 5 respectively.

EXAMPLE 5

[0066] A one-step fiber reinforced starch-vegetable oil graft copolymer reaction was carried out as follows:

[0067] 525 gm of granular corn-starch (Corn Products, Inc.) was first mixed with 15.7 gm maleic anhydride (Aldrich Chemical Company) and further mixed with 56 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride). Further, 175 gm glycerol mixed with 1.05 gm Luperox 101, was added, and the mixture was kneaded in a kitchen blender for 15 minutes. To this mixture was added 300 gm Kayocell (wood fiber) and further kneaded for 15 minutes. This mixture was extruded using CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40. The temperature profile and the extrusion processing conditions are shown in Table 2. The vent port was kept open to remove unreacted maleic acid and water. Screw configuration

consisting of both conveying and kneading elements was used and is shown in Figure 1. The extruded strand was palletized and stored in an oven at 100°C. The ESEM image of the blend shows good adhesion between the starch and the fiber and is shown in Figure 6. Injection molding conditions are shown in Table 3.

TABLE 3 Injection molding processing conditions in the synthesis of dog bone samples of fiber reinforced starch-vegetable oil graft copolymers

Temperature profile (All temperatures are in °F)

Nozzle	Zone 1	Zone 2	Zone 3
380	370	360	340
380	372	364	341

Oil temperature 104°F
Feed throat temperature 120°F
Hold pressure 800 psi
Pack pressure 1400 psi
Timer conditions
Inject high 10 sec
Pack 3 sec
Hold 3 sec
Cooling 45 sec
Extruder delay 16 sec
Open dwell 1 sec
Shot size 1 in
Shot velocity 0.3 in/s
Cushion 0.25 in
Transfer position 0.5 in
Mold temperature 125°F

Tensile properties of the injection molded articles were determined using INSTRON Mechanical Testing Equipment fitted

with a 1000 lbs load cell The crosshead speed was 1 inch per minute The samples were conditioned at 23°C and 50% Relative Humidity for 40 hours before being tested according to ASTM D-882 testing Tensile properties are depicted in Figure 8 and Figure 9

Example 6

[0068] A one-step fiber reinforced starch-vegetable oil graft copolymer reaction was carried out as follows

[0069] 400 gm of granular corn-starch (Corn Products, Inc) was first mixed with 12 gm maleic anhydride (Aldrich Chemical Company) and further mixed with 56 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further, 100 gm glycerol mixed with 0.8 gm Luperox 101, was added, and the mixture was kneaded in a kitchen blender for 15 minutes To this mixture was added 300 gm Kayocell (wood fiber) and 200 gm linear Polycaprolactone polymer (Dow Chemical (Midland, MI), TONE 787) and further kneaded for 15 minutes This mixture was extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40 The temperature profile and the extrusion processing conditions are shown in Table 2 The vent port was kept open to remove unreacted maleic acid and water Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1 The extruded strand was palletized and stored in an oven at 100°C The ESEM image of the blend is shown in Figure 7 Injection molding conditions are shown in Table 4

TABLE 4 Injection molding processing conditions in the synthesis of dog bone samples of fiber reinforced starch-vegetable oil graft copolymers containing polyester or modified polyester

Temperature profile (All temperatures are in °F)

Nozzle	Zone 1	Zone 2	Zone 3
370	360	350	340
369	359	353	341

Oil temperature 110°F
Feed throat temperature 126°F
Hold pressure 900 psi
Pack pressure 1300 psi

Timer conditions

Inject high 10 sec
Pack 3 sec
Hold 2 sec
Cooling 70 sec
Extruder delay 16 sec
Open dwell 1 sec
Shot size 1 05 in
Shot velocity 1 25 in/s
Cushion 0 25 in
Transfer position 0 5 in
Mold temperature 70°F

Tensile properties of the injection molded articles were determined using INSTRON Mechanical Testing Equipment fitted with a 1000 lbs load cell. The crosshead speed was 1 inch per minute. The samples were conditioned at 23°C and 50% Relative Humidity for 40 hours before being tested according to ASTM D-882 testing. Tensile properties are depicted in Figure 8 and Figure 9.

Example 7

[0070] A one-step fiber reinforced starch-vegetable oil graft copolymer reaction was carried out as follows

[0071] 400 gm of granular corn-starch (Corn Products, Inc) was first mixed with 12 gm maleic anhydride (Aldrich Chemical Company) and further mixed with 56 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further 100 gm glycerol mixed with 0.8 gm LUPEROX 101, was added, and the mixture was kneaded in a blender for 15 minutes To this mixture was added 300 gm Kayocell (wood fiber) and 200 gm Eastman Polyester (EPE) (Poly butylenes (adipate-co-terephthalate) obtained from Eastman Chemicals) and further kneaded for 15 minutes This mixture was extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40 The temperature profile and the extrusion processing conditions are shown in Table 2 The vent port was kept open to remove unreacted maleic acid and water Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1 The extruded strand was pelletized and stored in an oven at 100°C

Injection molding conditions are shown in Table 4 Tensile properties of the injection molded articles were determined using INSTRON Mechanical Testing Equipment fitted with a 1000 lbs load cell The cross head speed was 1 inch per minute The samples were conditioned at 23°C and 50% Relative Humidity for 40 hours before being tested according to ASTM D-882 testing Tensile properties are depicted in Figure 8 and Figure 9

EXAMPLE 8

[0072] A one-step fiber reinforced starch-vegetable oil graft copolymer reaction was carried out as follows

[0073] 400 gm of granular corn-starch (Corn Products, Inc)

was first mixed with 12 gm maleic anhydride (Aldrich Chemical Company) and further mixed with 56 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further, 100 gm glycerol mixed with 0.8 gm LUPEROX 101, was added, and the mixture was kneaded in a kitchen blender for 15 minutes To this mixture was added 300 gm Kayocell (wood fiber) and 400 gm linear Polycaprolactone polymer (Dow Chemical, TONE 787) obtained from Eastman Chemicals, Rochester, NY) and further kneaded for 15 minutes This mixture was extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40 The temperature profile and the extrusion processing conditions are shown in Table 2 The vent port was kept open to remove unreacted maleic acid and water Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1 The extruded strand was palletized and stored in an oven at 100°C Injection molding conditions are shown in Table 4 Tensile properties of the injection molded articles were determined using INSTRON Mechanical Testing Equipment fitted with a 1000 lbs load cell The cross head speed was 1 inch per minute The samples were conditioned at 23°C and 50% Relative Humidity for 40 hours before being tested according to ASTM D-882 testing Tensile properties are depicted in Figure 8 and Figure 9

EXAMPLE 9

[0074] A one-step fiber reinforced starch-vegetable oil graft copolymer reaction was carried out as follows

[0075] 1810 gm of granular corn-starch (Corn Products, Inc) was first mixed with 54.3 gm maleic anhydride (Aldrich Chemical Company) and further mixed with 127 gm of maleated corn oil (1 mole of maleated corn oil contains one mole of maleic anhydride) Further, 603 gm glycerol mixed with 3.62 gm

LUPEROX 101, was added, and the mixture was kneaded in a kitchen blender for 15 minutes. To this mixture was added 1088 gm SOLKA FLOX (Cellulose fiber, obtained from International Fiber Corporation) and further kneaded for 16 minutes. This mixture was extruded using a CENTURY twin-screw co-rotating extruder with a screw diameter of 30 mm and a L/D of 40. The temperature profile and the extrusion processing conditions are shown in Table 2. The vent port was kept open to remove unreacted maleic acid and water. Screw configuration consisting of both conveying and kneading elements was used and is shown in Figure 1. The extruded strand was palletized and stored in an oven at 100°C. Injection molding conditions are shown in Table 5.

TABLE 5 Injection molding processing conditions in the synthesis of dog bone samples of cellulose fiber (SOLKA FLOC) reinforced starch-vegetable oil graft copolymers

Temperature profile (All temperatures are in °F)

Nozzle	Zone 1	Zone 2	Zone 3
380	370	360	340
376	373	366	341

Oil temperature 110°F

Feed throat temperature 126°F

Hold pressure 800 psi

Pack pressure 1400 psi

Timer conditions

Inject high 10 sec

Pack 3 sec

Hold 3 sec

Cooling 45 sec

Extruder delay 16 sec
Open dwell 2 sec
Shot size 1 in
Shot velocity 0.7 in/s
Cushion 0.25 in
Transfer position 0.5 in
Mold temperature 130°F

Tensile properties of the injection molded articles were determined using INSTRON Mechanical Testing Equipment fitted with a 1000 lbs load cell. The crosshead speed was 1 inch per minute. The samples were conditioned at 23°C and 50% Relative Humidity for 40 hours before being tested according to ASTM D-882 testing. Tensile properties are depicted in Figure 8 and Figure 9.

[0076] The above examples demonstrate that the present invention provides new starch-vegetable oil based graft copolymers and their fiber reinforced composites which utilize agricultural resources that is biobased and biodegradable. The polymeric materials made in accordance with the present invention is sustainable and environmentally compatible, this being achieved by designing and engineering fully biodegradable materials that are thermoplastic yet breakdown under appropriate environmental conditions in a manner similar to their lignocellulosic counterparts.

[0077] It is intended that the foregoing description be only illustrative of the present invention and that the present invention be limited only by the hereinafter appended claims.

WE CLAIM

- 1 A biodegradable starch-vegetable oil graft copolymer composition which is a reactively blended mixture comprising a granular starch, a vegetable oil or a derivative thereof comprising a modifier reactive with the starch, and a hydroxylated plasticizer
- 2 The composition of Claim 1 wherein the granular starch is selected from the group consisting of cornstarch, potato starch, tapioca starch, rice starch, wheat starch, cassava starch and high amylase corn starch
- 3 The composition of Claim 1 wherein the starch in the mixture is 5% to 90% by weight
- 4 The composition of Claim 1 wherein the starch has a moisture content of about 0.5% to 15% by weight of the starch
- 5 The composition of Claim 1 wherein the starch is in a pulverized form
- 6 The composition of Claim 1 wherein the starch is chemically modified with the modifier
- 7 The composition of Claim 1 wherein the vegetable oil is a derivative which is epoxylated, hydroxylated or maleated
- 8 The composition of Claim 1 wherein the vegetable oil is selected from the group consisting of corn oil, soybean oil, maleated soybean oil and epoxidized soybean oil and mixtures thereof

- 9 The composition of Claim 1 wherein the vegetable oil is 1% to 10% by weight of the composition
- 10 The composition of Claim 1 wherein the plasticizer is a polyhydric alcohol in an amount between 10 to 40 wt-% of the total of the composition
- 11 The composition of Claim 1 wherein a free-radical peroxide initiator is provided in the mixture
- 12 The composition of Claim 11 wherein the free-radical initiator is 2,5-Bis(tert-butylperoxy)-2,5-dimethylhexane (LUPEROX 101)
- 13 The composition of Claim 11 wherein a free radical peroxide initiator is provided in the mixture in an amount between about 0.01 to 3-wt% of the total weight of the composition
- 14 The composition of Claim 1 further comprising a fiber
- 15 The composition of Claim 14 wherein the fiber is selected from the group consisting of cotton, wood, flax, abaca, sisal, ramie, hemp, and bagasse
- 16 The composition of Claim 15 wherein the fiber has an aspect ratio of at least about 10:1
- 17 The composition of Claim 12 wherein the fiber is present in an amount between about 3% to 80% by weight of the composition

- 18 The composition of Claim 1 comprising the modifier which is selected from the group consisting of cyclic or dibasic acid and anhydrides thereof
- 19 The composition of Claim 18 wherein the modifier is present in the composition in amount of about 0.1 to about 10% by weight of the starch
- 20 The composition of Claim 1 further comprising a biodegradable polyester selected from the group consisting of poly(caprolactone) (PCL), poly(vinylacetate-co-vinylalcohol) (PVAc/VA), poly(lactic acid) or polylactide (PLA), poly(glycolic acid) or polyglycolide (PGA), poly(beta-hydroxyalkanoates) (PHA), and aliphatic aromatic copolyesters
- 21 The composition of Claim 20 wherein the biodegradable polyester is 1% to 60% by weight of the composition
- 22 A process for producing a biodegradable starch vegetable oil copolymer composition which comprises
(a) reactively blending a mixture of a granular starch, a vegetable oil or a derivative thereof comprising a modifier reactive with the starch, and a plasticizer in a reactor, and
(b) removing the composition from the reactor to produce the composition
- 23 The process of Claim 22 wherein the reactor is an extruder

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- 24 The process of Claim 22 wherein the vegetable oil derivative is a maleated vegetable oil
- 25 The process of Claim 22 wherein the mixture further comprises a fiber
- 26 The process of Claim 22 wherein the plasticizer is a polyhydric alcohol

ABSTRACT

A new starch-vegetable oil graft copolymer, wherein the vegetable oil has been reacted onto the starch backbone using thermal or free radical initiators has been produced in a twin-screw co-rotating extruder. The starch-vegetable oil graft copolymer can further be reinforced with biofiber in the presence of an optional modifier such as maleic anhydride by reactive extrusion processing to form composites suitable to be injection molded into biodegradable articles.

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Docket No
MSU 4.1-726

Declaration and Power of Attorney For Patent Application

English Language Declaration

As a below named inventor, I hereby declare that

My residence post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (If only one name is listed below) or an original, first and joint inventor (If plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

STARCH-VEGETABLE OIL GRAFT COPOLYMERS AND THEIR BIOFIBER COMPOSITES, AND A PROCESS FOR THEIR MANUFACTURE

the specification of which
(check one)

☒ is attached hereto

☐ was filed on _____ as United States Application No. or PCT International
Application Number _____
and was amended on _____

(If applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or (f), or 365(b) of any foreign application(s) for patent, inventor's or plant breeder's rights certificate(s), or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent, inventor's or plant breeder's rights certificate(s), or any PCT international application having a filing date before that of the application on which priority is claimed

Prior Foreign Application(s)

Priority Not Claimed

_____ (Number)	_____ (Country)	_____ (Day/Month/Year Filed)	☐
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I hereby claim the benefit under 35 U S C Section 119(a) of any United States provisional application(s) listed below

_____ (Application Serial No)	_____ (Filing Date)
_____ (Application Serial No.)	_____ (Filing Date)
_____ (Application Serial No)	_____ (Filing Date)

I hereby claim the benefit under 35 U S C Section 120 of any United States application(s), or Section 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U S C Section 112, I acknowledge the duty to disclose to the United States Patent and Trademark Office all information known to me to be material to patentability as defined in Title 37, C F R , Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application

_____ (Application Serial No)	_____ (Filing Date)	_____ (Status) (patented pending, abandoned)
_____ (Application Serial No.)	_____ (Filing Date)	_____ (Status) (patented, pending abandoned)
_____ (Application Serial No)	_____ (Filing Date)	_____ (Status) (patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon

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POWER OF ATTORNEY As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith (list name and registration number)

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Fourth inventor's signature	Date
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Citizenship	
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Full name of fifth inventor if any	
Fifth inventor's signature	Date
Residence	
Citizenship	
Post Office Address	

Full name of sixth inventor if any	
Sixth inventor's signature	Date
Residence	
Citizenship	
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**VERIFIED STATEMENT (DECLARATION) CLAIMING
SMALL ENTITY STATUS - NONPROFIT ORGANIZATION**

Docket No.
MSU 4.1-726

Serial No.

Filing Date

Patent No.

Issue Date

Applicant/ Patentee: **Ramani Narayan, Sunder Balakrishnan and Bee-Young Shin**

Invention **STARCH-VEGETABLE OIL GRAFT COPOLYMERS AND THEIR BIOFIBER COMPOSITES, AND
A PROCESS FOR THEIR MANUFACTURE**

I hereby declare that I am an official empowered to act on behalf of the nonprofit organization identified below

NAME OF ORGANIZATION Board of Trustees of Michigan State University

ADDRESS OF ORGANIZATION 236 Administration Building
East Lansing, Michigan 48824

TYPE OF NONPROFIT ORGANIZATION

- ☒ University or other Institute of Higher Education
- ☐ Tax Exempt under Internal Revenue Service Code (26 U.S.C. 501(a) and 501(c)(3))
- ☐ Nonprofit Scientific or Educational under Statute of State of The United States of America
Name of State: Citation of Statute:
- ☐ Would Qualify as Tax Exempt under Internal Revenue Service Code (26 U.S.C. 501(a) and 501(c)(3)) if Located in The United States of America
- ☐ Would Qualify as Nonprofit Scientific or Educational under Statute of State of The United States of America if Located in The United States of America
Name of State: Citation of Statute:

I hereby declare that the above-identified nonprofit organization qualifies as a nonprofit organization as defined in 37 CFR 1.27(a)(3) for purposes of paying reduced fees to the United States Patent and Trademark Office under section 41(a) and (b) of Title 35, United States Code regarding the invention described in.

- ☒ the specification to be filed herewith.
- ☐ the application identified above
- ☐ the patent identified above

I hereby declare that rights under contract or law have been conveyed to and remain with the nonprofit organization with regard to the above identified invention

If the rights held by the above-identified nonprofit organization are not exclusive, each individual, concern or organization having rights to the invention is listed on the next page and no rights to the invention are held by any person, other than the inventor, who could not qualify as a small entity person under 37 CFR 1.27(a)(1) or by any concern which would not qualify as a small business concern under 37 CFR 1.27(a)(2) or a nonprofit organization under 37 CFR 1.27(a)(3)

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- ☒ no such person, concern or organization exists
☐ each such person, concern or organization is listed below

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☐ Individual ☐ Small Business Concern ☐ Nonprofit Organization

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FULL NAME
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Separate verified statements are recommended from each named person, concern or organization having rights to the invention averring to their status as small entities.

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.27(g)(2))

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

NAME OF PERSON SIGNING

Loraine J. Hudson

TITLE IN ORGANIZATION

Director of Intellectual Property

ADDRESS OF PERSON SIGNING

Michigan State University
238 Administration Building
East Lansing, Michigan 48824

SIGNATURE

Loraine J. Hudson

DATE

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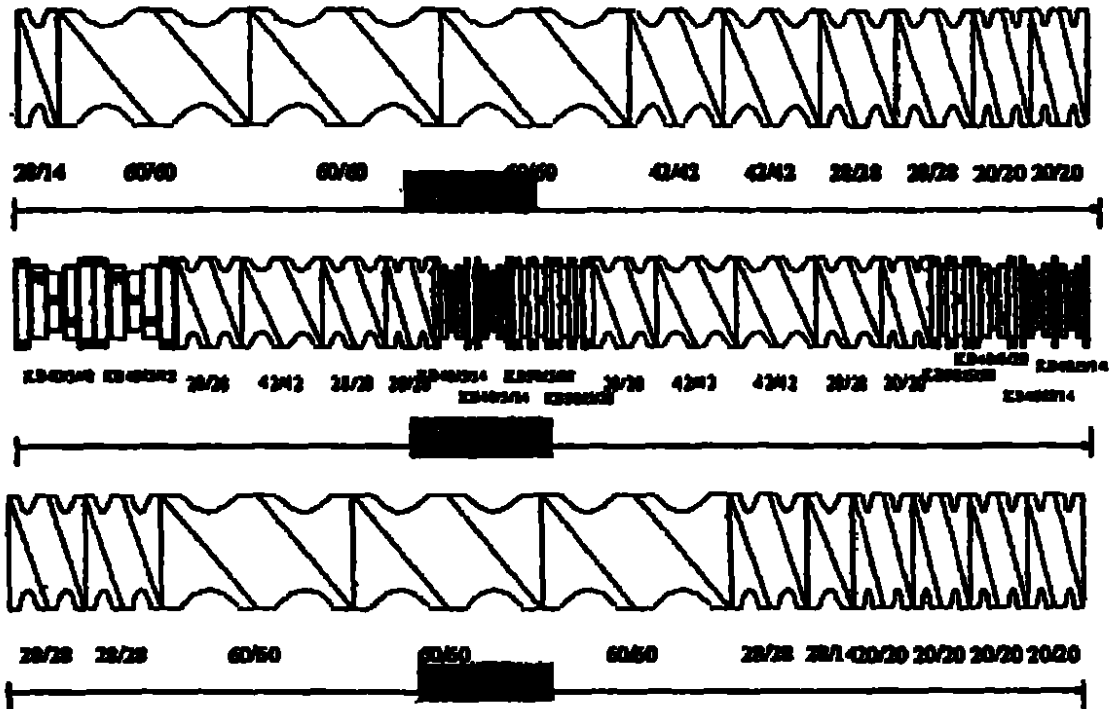
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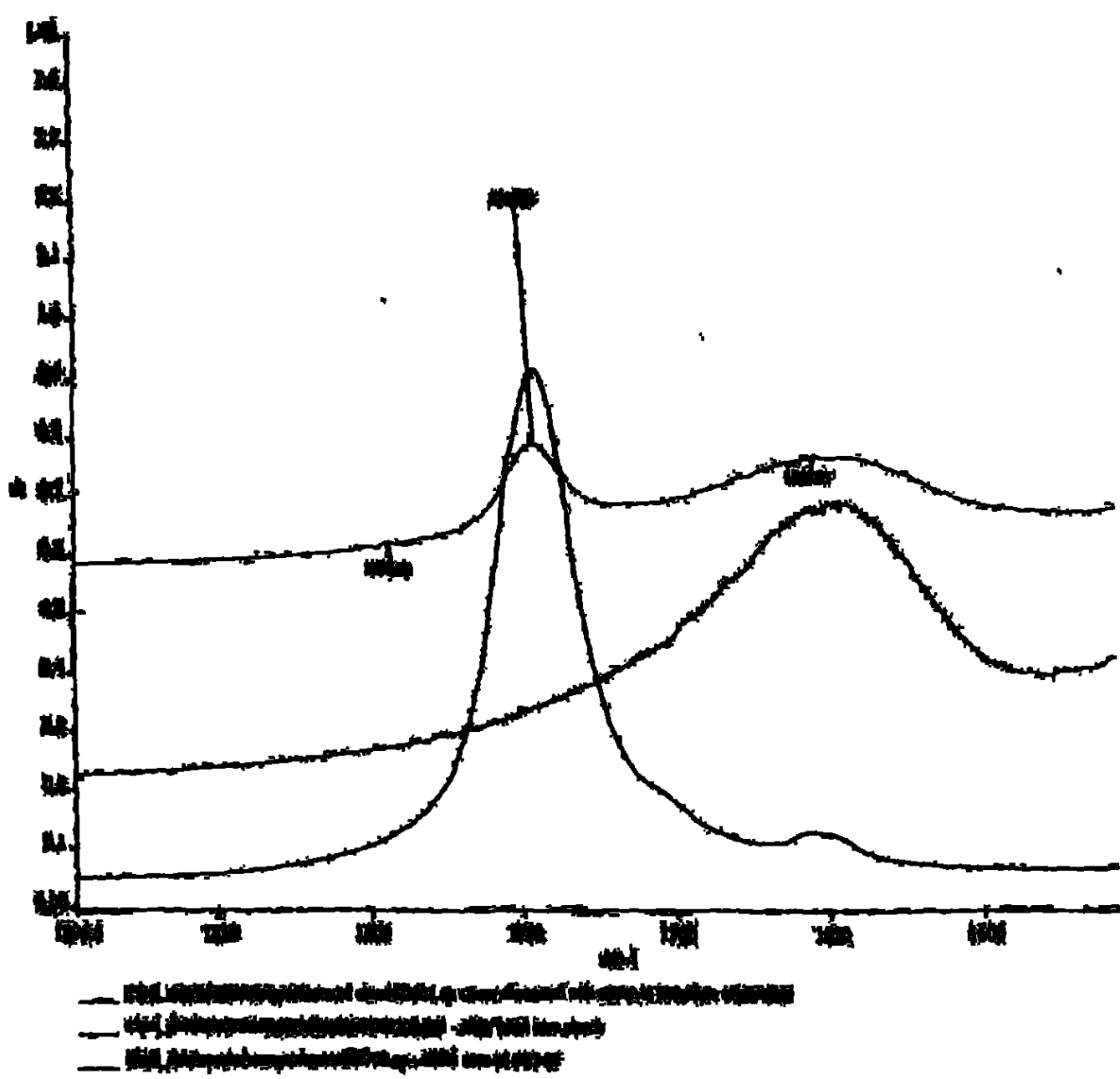
FIGURE 1 Screw Configuration used in the synthesis of starch-vegetable oil graft copolymers by reactive extrusion processing.



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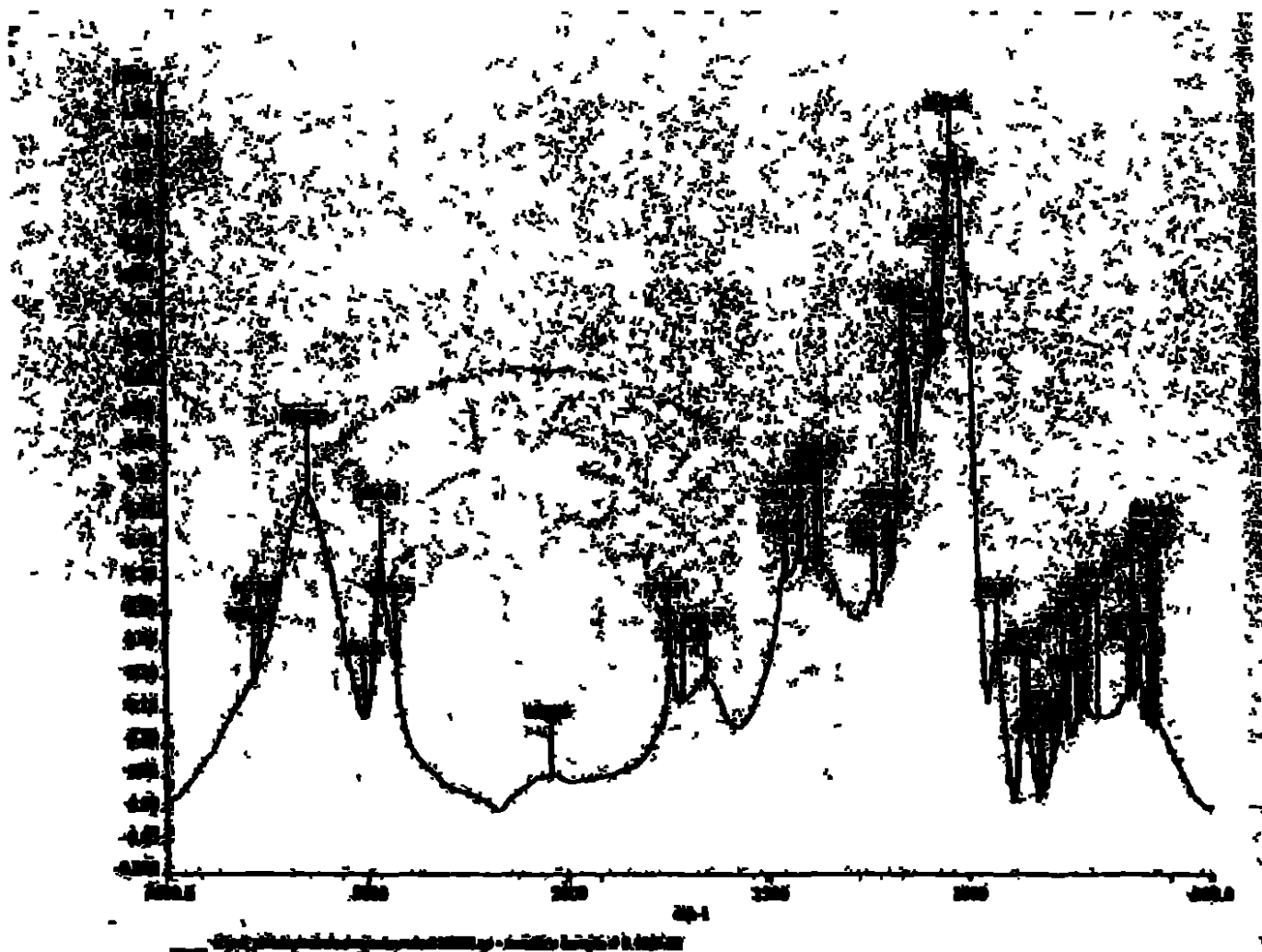
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FIGURE 2 FTIR scan of extracted product confirming reactivity between starch and corn oil in the synthesis of starch-corn oil graft copolymers



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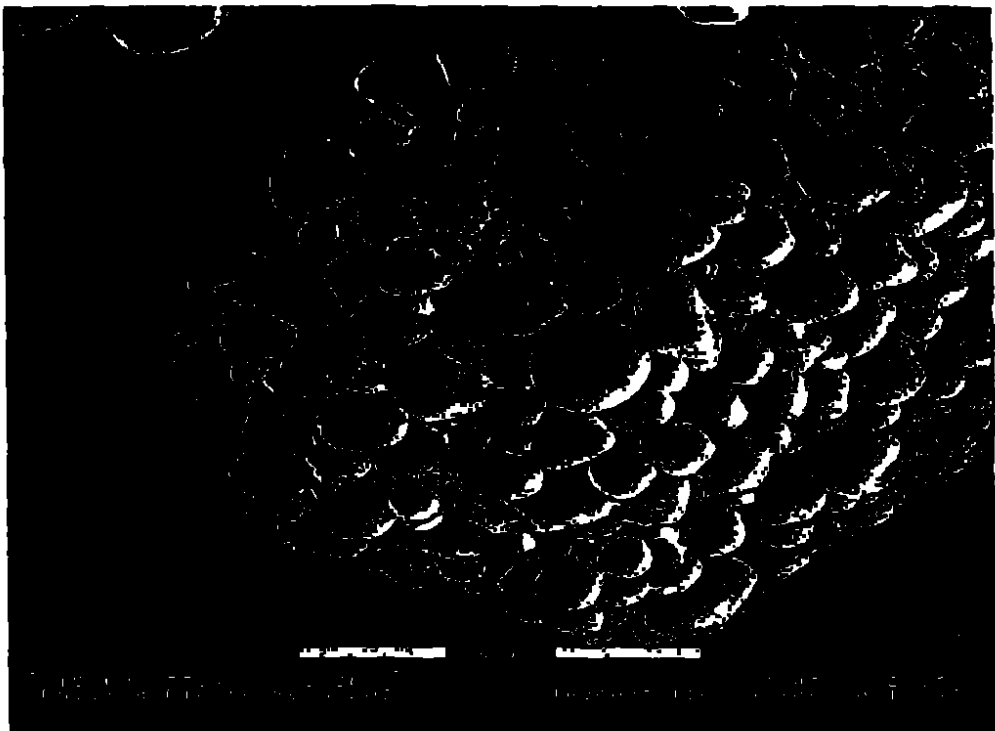
FIGURE 3 FTIR scan of extracted product confirming reactivity between starch and maleated corn oil in the synthesis of starch-corn oil graft copolymers (absence of free-radical initiator)



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FIGURE 4 ESEM Image of regular granular starch



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FIGURE 5 ESEM Image of starch-maleated corn oil graft copolymer produced by reactive extrusion processing.



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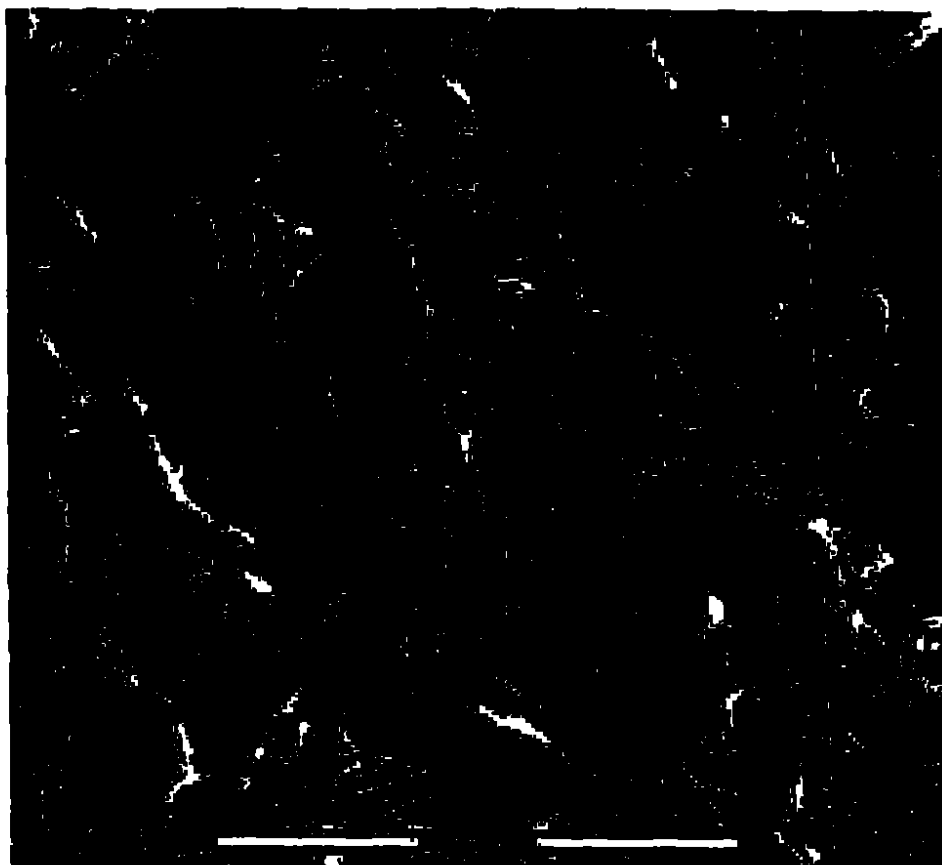
FIGURE 6 ESEM image of fiber reinforced starch-vegetable oil graft copolymer



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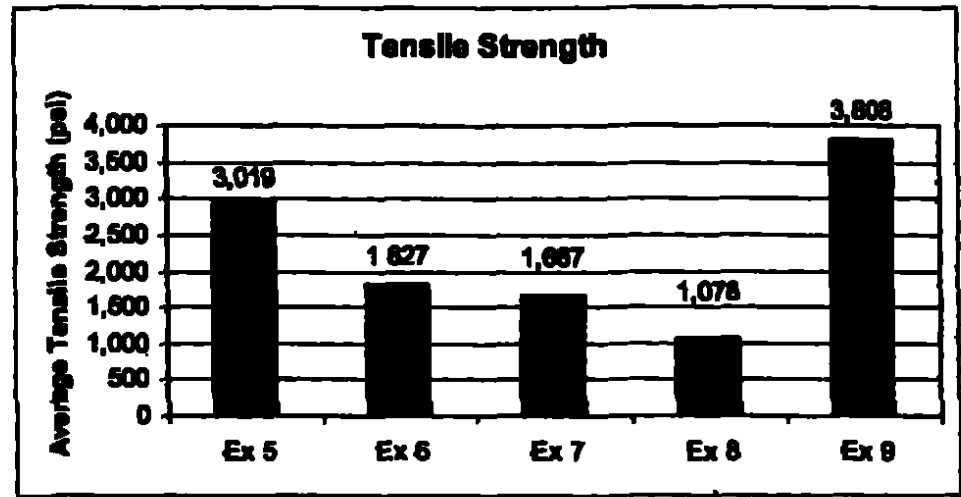
FIGURE 7 ESEM image of fiber reinforced starch-vegetable oil graft copolymers containing Polycaprolactone polyester



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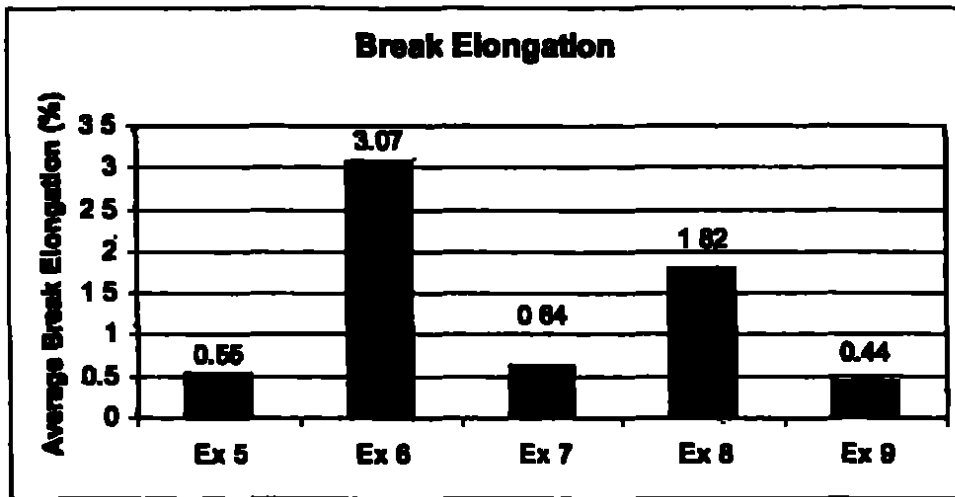
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FIGURE 8 Tensile Strength properties of various fiber re-inforced starch-vegetable oil graft copolymer blends as explained in Examples 5-9



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FIGURE 9 Break elongation properties of various fiber re-inforced starch-vegetable oil graft copolymer blends as explained in Examples 5-9



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Apoderado(a) y/o Representante de
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Teniendo en cuenta que la solicitud de privilegio de patente que se tramita bajo el expediente indicado en la referencia, reúne los requisitos de forma establecidos en los artículos 26 y 27 de la Decisión 486 de la Comisión de la Comunidad Andina, y en las demás disposiciones vigentes sobre la materia, se envía el extracto de la solicitud a la Oficina de Comunicaciones para efecto de su publicación en la Gaceta de Propiedad Industrial, conforme lo establece el artículo 40 de la misma Decisión

Siendo manifestación expresa del interesado de efectuar la publicación "inmediato", está se realizará en la próxima gaceta de Propiedad Industrial

Cúmplase
Dado en Bogotá DC,

14 MAY 2007

ALIX CARMENZA CESPEDES DE VERGEL
Jefe de la división de Nuevas Creaciones

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