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SUPERINDUSTRIA Y COMERCIO
*Radicación : 00029095 00000004 Folios: 17
Fecha (AMD): 2003-01-22 17:17:20 5n
Trámite : 002 PATENTES D 1 REGISTRO/ 329 COMPLETOS
Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

**FORMULARIO UNICO DE CORRECCIONES Y MODIFICACIONES
2000-10**

1	SOLICITUD DE:	
☐	Corrección de Error Material	☒ Modificación a una solicitud
2 SOLICITANTE	Nombre: DOW GLOBAL TECHNOLOGIES, INC. sociedad constituida y existente bajo las leyes del Estado de Delaware, Estados Unidos de América. Dirección: 1790 Building, Washington Street, Midland, Michigan 48676, Estados Unidos de América. Teléfono: 546 09 70 E-mail: clientes@camyp.com Fax: 249 40 70 Domicilio: Midland, Michigan, Estados Unidos de América	IDENTIFICACION C.C. ☐ NIT ☐ C.E. ☐ Otro ☐ Número: _____
3 REPRESENTANTE O APODERADO	Nombre: Emilio FERRERO Dirección: Carrera 4ª No. 72-35 Teléfono: 347 36 11 E-mail: cavelier@cavelier.com Fax: 217 92 11 Domicilio: Bogotá, Colombia	IDENTIFICACION C.C. ☒ NIT ☐ C.E. ☐ Otro ☐ Número: <u>438.019</u> TP <u>31.929</u>
4	Número de expediente: <u>00.029.095</u> Clase: <u>A61K 047038</u> Título del Invento: CÁPSULAS DE ÉTER DE CELULOSA LIBRES DE TURBIEDAD Y PROCESO PARA HACER LAS MISMAS	
5	Descripción de la corrección o modificación solicitada: La sociedad THE DOW CHEMICAL COMPANY, solicitante de la patente objeto de este expediente, ha traspasado sus derechos a la sociedad DOW GLOBAL TECHNOLOGIES, INC. Por lo tanto pido a usted expedir el certificado de patente a favor de DOW GLOBAL TECHNOLOGIES, INC.	
6	Comprobante de pago No. <u>03 - 2,484</u> Fecha: <u>14 de enero de 2003</u>	
7	Anexos: ☒ Comprobante de pago de la tasa por \$ 82.000.00 ☒ Poderes, si fuere el caso ☒ Certificado de existencia y representación legal, cuando el solicitante sea persona jurídica	
8	NOMBRE: <u>EMILIO FERRERO</u> FIRMA:  C.C. 438.019 de Usaquén T.P.A. 31.929	

[illegible]

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74

(TRANSFER)

-- SEE ATTACHED SCHEDULE --

ASSIGNEE

Notario Sexto del Circulo
DE BOGOTA, MAGO CONSTAR QUE LA
FOTOCOPIA COINCIDE CON EL ORIGINAL
QUE HE TENIDO A LA VISTA

15 ENF 2003

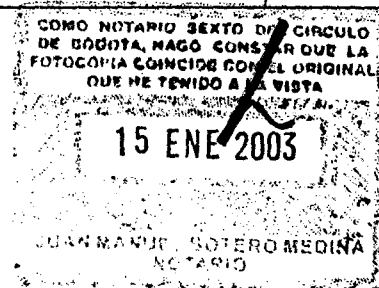
JUAN MANUEL BOTERO MEDINA
NOTARIO

Attach Notarial Certificate (Corporation)

APPENDIX TO TRANSFER ASSIGNMENT

80
75

Country	Filing number	Filing date	Title (English)	Invention file N°
COLOMBIA	00029095	April 19, 2000	HAZE-FREE CELLULOSE ETHER CAPSULES AND PROCESS FOR MAKING	44243
COLOMBIA	00032228	May 5, 2000	AN IMPROVED PROCESS FOR PRODUCING HIGH MOLECULAR WEIGHT MONOVINYLDENE AROMATIC POLYMERS AND POLYMODAL COMPOSITIONS	44538
COLOMBIA	00035460	May 16, 2000	BIMODAL RUBBERS AND RUBBER MODIFIED HIGH IMPACT MONOVINYLDENE AROMATIC POLYMERS PRODUCED THEREFROM	44594
COLOMBIA	00051051	July 7, 2000	PROCESS FOR POLYOXIDES INITIATED WITH ACETYLENIC ALCOHOLS	60068
COLOMBIA	00051057	July 7, 2000	PROCESS FOR FRACTIONATION OF TERTIARY ALCOHOL INITIATED POLYOLS WITH CONCOMITANT REMOVAL OF DOUBLE METAL CYANIDE CATALYST	60069
COLOMBIA	00095529	December 15, 2000	MONOVINYLDENE AROMATIC POLYMERS WITH IMPROVED PROPERTIES AND A PROCESS FOR THEIR PREPARATION	60395A
COLOMBIA	01007465	February 1, 2001	A PROCESS FOR PRODUCING THERMOFORMABLE FOAM SHEET USING A PHYSICAL BLOWING AGENT	60380A
COLOMBIA	391235	June 11, 1993	A FOAM STABILIZING ADDITIVE AND POLYURETHANE FOAM PREPARED IN THE PRESENCE THEREOF	40254
COLOMBIA	96051615	September 27, 1996	CROSSLINKED POLYOLEFINIC FOAMS WITH ENHANCED PHYSICAL PROPERTIES AND A DUAL CURE PROCESS OF PRODUCING SUCH FOAMS	42505A
COLOMBIA	97045591	August 8, 1997	3-HETEROATOM SUBSTITUTED CYCLOPENTADIENYL-CONTAINING METAL COMPLEXES AND OLEFIN POLYMERIZATION PROCESS	42964A
COLOMBIA	98002573	January 21, 1998	POLYMERS CONTAINING HIGHLY GRAFTED RUBBERS	43122A
COLOMBIA	98036383	June 25, 1998	ACID CATALYZED POLYMERIZATION	43168A
COLOMBIA	98046530	August 13, 1998	HIGH GLOSS HIGH IMPACT MONOVINYLDENE AROMATIC POLYMERS	43396A
COLOMBIA	99036208	June 9, 1999	CENTRIFUGAL METHOD AND APPARATUS FOR DEVOLATILIZING POLYMERS	44240A
COLOMBIA	99062023	September 30, 1999	HIGH EFFICIENCY GEAR PUMP FOR PUMPING HIGHLY VISCOUS FLUIDS	44104
COLOMBIA	99081619	December 30, 1999	HIGH IMPACT MONOVINYLDENE AROMATIC POLYMERS	44331



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: **Ann Heritier**

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

4. bears the seal of: **Ann Heritier**

Bay County, Michigan

CERTIFIED:

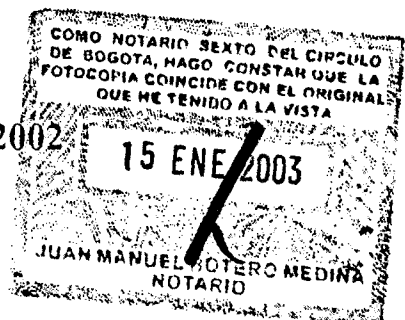
5. at Lansing, Michigan

6. the 31st of October, 2002

7. by Secretary of State, State of Michigan

8. NO. 28762F-02

9. Seal/Stamp:



10. Signature:

Candice S. Miller

Candice S. Miller

This certification attests only to the authenticity of the signature of the official who signed the affixed 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.

This Notarial Certificate 550 is in the ENGLISH language and has the same meaning in other languages with the same 550 number.

FORM 550

NOTARIAL CERTIFICATE (Corporation)

UNITED STATES OF AMERICA)
STATE OF Michigan) SS
County/Parish of Midland)

On this 21 day of October, 2002, personally appeared before me,
Graham E. Taylor, Associate General Counsel, Intellectual Property,
The Dow Chemical Company
known or identified to me to be the individual described in and who executed the foregoing document

Transfer Assignment for an on behalf of The Dow Chemical Company

and acknowledged the same as a free act and deed for uses and purposes therein expressed by authority of the said Corporation incorporated under the laws of the State of Delaware, United States of America.

I further certify that said authority, dated **September 7, 1999**, is based on a **Resolution of the Board of Directors**, which I have seen, appointing the said individual **Attorney-in-fact** with full power to sign the foregoing document on behalf of the said corporation and that the purposes for which said document is granted are within the scope of the objects or activities of said corporation.

SEAL



Notary Public
ANN HERITIER
NOTARY PUBLIC, BAY COUNTY, MICHIGAN
ACTING IN MIDLAND COUNTY, MICHIGAN
MY COMMISSION EXPIRES DECEMBER 3, 2002

NOTARIAL CERTIFICATE (Corporation)

UNITED STATES OF AMERICA)
STATE OF Michigan) SS
County/Parish of Midland)

On this 21 day of October, 2002, personally appeared before me, **Michael L. Glenn, Secretary, DOW GLOBAL TECHNOLOGIES INC.,** known or identified to me to be the individual described in and who executed the foregoing document

Transfer Assignment for an on behalf of Dow Global Technologies Inc.

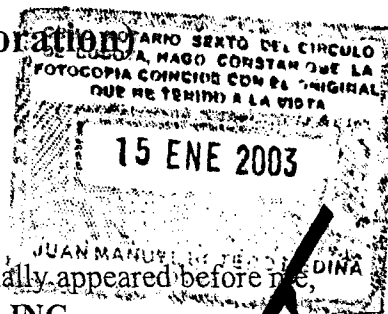
and acknowledged the same as a free act and deed for uses and purposes therein expressed by authority of the said Corporation incorporated under the laws of the State of Delaware, United States of America.

I further certify that said authority, dated **February 11, 2002**, is based on an **Authorization Policy of the said Corporation**, which I have seen, appointing the said individual **Attorney-in-fact** with full power to sign the foregoing document on behalf of the said corporation and that the purposes for which said document is granted are within the scope of the objects or activities of said corporation.

SEAL



Notary Public
ANN HERITIER
NOTARY PUBLIC, BAY COUNTY, MICHIGAN
ACTING IN MIDLAND COUNTY, MICHIGAN
MY COMMISSION EXPIRES DECEMBER 3, 2002



TRADUCCIÓN No. 07.01/03 realizada por María Elvira Martelo,
traductora oficial aprobada por el Ministerio de Justicia
mediante Resolución No. 2879 de 1995 y reconocida por el
Ministerio de Relaciones Exteriores.

Este traspaso 990 es en inglés y tiene el mismo significado en otros idiomas con el mismo número 990

☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐ ☐

Este traspaso 990 es en inglés y tiene el mismo significado en otros idiomas con el mismo número 990

TRASPASO

Nosotros, **CEDENTE:** THE DOW CHEMICAL COMPANY

De: 2030 Dow Center

Midland, Michigan 48674

Estados Unidos de América

Una sociedad organizada y existente bajo las leyes del Estado de Delaware, Estados Unidos de América, por el presente reivindicamos y declaramos que somos los propietarios registrados de la(s) siguiente(s) solicitud(es) de patente y de otros derechos y facultades obtenibles o que se pueden ejercer con respecto a las mismas según lo enumerado en la(s) solicitud(es) de patente o patente(s) COLOMBIANAS:

-VÉASE EL ANEXO-

por el presente, por una contraprestación
recibida, vendemos, cedemos y transferimos a:

Nosotros, **CEDENTE:** DOW GLOBAL TECHNOLOGIES, INC.

De: Washington Street

1790 Building

Midland, Michigan 48674

Estados Unidos de América

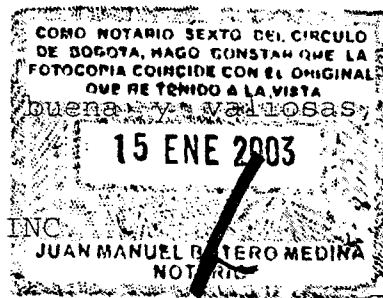
Una sociedad organizada y existente bajo las leyes del Estado de Delaware, Estados Unidos de América, todos nuestros derechos, títulos e intereses en y a dicha(s) solicitud(es) de patente o patente(s) y consentimos al registro de este Traspaso

CEDENTE

Suscrito y jurado por nosotros:
En Midland, Michigan 48674, EUA

CESIONARIO

Aceptado por nosotros:
En Midland, 48674, USA



Hoy 21 de octubre de 2002
 THE DOW CHEMICAL COMPANY
 Por: (firma ilegible)
 Graham E. Taylor
 Asesor General Asociado
 Propiedad Intelectual
 SELLO
 CORPORATIVO

Hoy 21 de octubre de 2002
 DOW GLOBAL TECHNOLOGIES, INC.
 Por: (firma ilegible)
 Michael L. Glenn
 Secretario
 SELLO
 CORPORATIVO

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APÉNDICE DEL TRASPASO

País	No. Present.	Fecha Present.	Título (Inglés)	No.Exp. Invento
COLOMBIA	00029095	Abril 19, 2000	CAPSULAS DE ÉTER DE CELULOSA LIBRES DE OPACIDAD	44243
COLOMBIA	00032228	Mayo 5, 2000	PROCESO MEJORADO PARA PRODUCIR POLÍMEROS AROMÁTICOS DE MONOVINILIDENO DE ALTO PESO MOLECULAR Y COMPOSICIONES POLIMODALES	44538
COLOMBIA	00035460	Mayo 16, 2000	CAUCHOS BIMODALES Y POLÍMEROS AROMÁTICOS DE MONOVINILIDENO DE ALTO IMPACTO MODIFICADOS CON CAUCHO PRODUCIDOS A PARTIR DE LOS MISMOS	44594
COLOMBIA	00051051	Julio 7, 2000	PROCESO PARA POLIOXIDOS INICIADOS CON ALCOHOLES ACETILÉNICOS	60068
COLOMBIA	00051057	Julio 7, 2000	PROCESO PARA FRACCIONAMIENTO DE POLIOLES INICIADOS CON ALCOHOL TERCIARIO CON ELIMINACIÓN DE CATALIZADOR METAL DOBLE	60069
COLOMBIA	00095529	Diciembre 15, 2000	POLÍMEROS AROMÁTICOS DE MONOVINILIDENO CON PROPIEDADES MEJORADAS Y UN PROCESO PARA SU PREPARACIÓN	60395A
COLOMBIA	01007465	Febrero 1, 2001	PROCESO PARA PRODUCIR LAMINAS DE ESPUMA TERMOFORMABLES UTILIZANDO UN AGENTE DE SOPLADO FÍSICO	60380A
COLOMBIA	391235	Junio 11, 1993	ADITIVO ESTABILIZADOR DE ESPUMA Y ESPUMA DE POLIURETANO PREPARADA EN PRESENCIA DEL MISMO	40254
COLOMBIA	96051615	Septiembre 27, 1996	ESPUMAS POLIOLEFINICAS DEGRADADAS CON PROPIEDADES FÍSICAS MEJORADAS Y UN PROCESO DE CURADO DUAL PARA PRODUCCIÓN DE DICHAS ESPUMAS	42505A
COLOMBIA	97045591	Agosto 8, 1997	COMPLEJOS METÁLICOS CON CICLOPENTADIENILO 3-HETEROÁTOMO SUSTITUIDO Y PROCESO DE POLIMERIZACIÓN DE OLEFINAS	42964A
COLOMBIA	98002573	Enero 21, 1998	POLÍMEROS CON CAUCHOS ALTAMENTE INJERTADO	43122A



COLOMBIA	98036383	Junio 25, 1998	POLIMERIZACION ACIDA CATALIZADA	43168A
COLOMBIA	98046530	Agosto 13, 1998	POLIMEROS AROMATICOS DE MONOVINILIDENO DE ALTO IMPACTO Y ALTO BRILLO	43396A
COLOMBIA	99036208	Junio 9, 1999	METODO CENTRIFUGO Y APARATO PARA DESVOLATILIZAR POLIMEROS	44240A
COLOMBIA	99062023	Septiembre 30, 1999	BOMBA DE ENGRANAJES DE ALTA EFICIENCIA PARA BOMBEAR FLUIDOS DE ALTA VISCOSIDAD	44104
COLOMBIA	99081619	Diciembre 30, 1999	POLIMEROS AROMATICOS DE MONOVINILIDENO DE ALTO IMPACTO	44331

CERTIFICADO NOTARIAL (Sociedad)

ESTADOS UNIDOS DE AMÉRICA

ESTADO DE Michigan SS

Condado/Parroquia de Midland

Hoy 21 de octubre de 2002, compareció personalmente ante mí
Graham E. Taylor, Asesor General Asociado, Propiedad
Intelectual, The Dow Chemical Company

Conocido o identificado por mí como el individuo descrito en, y
 que firmó el documento anterior

Traspaso por y a nombre de The Dow Chemical Company

Y reconoció el mismo como un acto libre para los usos y efectos
 allí expresados por autoridad de dicha Sociedad constituida bajo
 las leyes del Estado de Delaware, Estados Unidos de América

Además certifico que dicha autoridad, de fecha 7 de septiembre
 de 1999, se fundamenta en una Resolución de la Junta Directiva
 que he visto, nombrando a dicho individuo como Apoderado con
 plenas facultades para firmar el documento anterior a nombre de
 dicha sociedad y que los efectos para los cuales se otorga dicho
 documento se encuentran dentro del alcance de los objetos o
 actividades de dicha sociedad.

(firmado) Ann Heritier, Notario Público

(Sello en tinta) ANN HERITIER, CONDADO DE BAY, MICHIGAN, EN
 EJERCICIO EN EL CONDADO DE MIDLAND, MICHIGAN. MI COMISIÓN EXPIRA
 EL 3 DE DICIEMBRE DE 2002.

CERTIFICADO NOTARIAL (Sociedad)

ESTADOS UNIDOS DE AMÉRICA

ESTADO DE Michigan

SS

Condado/Parroquia de Midland

Hoy 21 de octubre de 2002, compareció personalmente ante mí Michael L. Glenn, Secretario, DOW GLOBAL TECHNOLOGIES, INC., Conocido o identificado por mí como el individuo descrito en, y que firmó el documento anterior

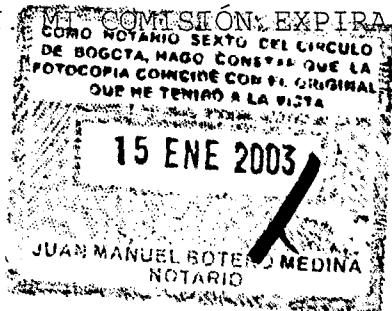
Traspaso por y a nombre de Dow Global Technologies, Inc.

Y reconoció el mismo como un acto libre para los usos y efectos allí expresados por autoridad de dicha Sociedad constituida bajo las leyes del Estado de Delaware, Estados Unidos de América.

Además certifico que dicha autoridad, de fecha 11 de febrero de 2002, se fundamenta en una Autorización de dicha Sociedad, la cual he visto, nombrando a dicho individuo como Apoderado con plenas facultades para firmar el documento anterior a nombre de dicha sociedad y que los efectos para los cuales se otorga dicho documento se encuentran dentro del alcance de los objetos o actividades de dicha sociedad.

(firmado) Ann Heritier, Notario Público

(Sello en tinta) ANN HERITIER, CONDADO DE BAY, MICHIGAN, EN EJERCICIO EN EL CONDADO DE MIDLAND, MICHIGAN. EL 3 DE DICIEMBRE DE 2002.



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:

Ann Heritier

3. Actuando en su capacidad de:

Notario Público de Michigan

4. lleva el sello/timbre de:

Ann Heritier

Condado de Bay, Michigan

CERTIFICADO

5. En Lansing, Michigan 6. el 31 de octubre de 2002
7. Por el Secretario de Estado, Estado de Michigan
8. No. 28762F-02
9. Sello/Timbre: El Gran Sello del Estado de Michigan
10. Firma: (Firmado) Candice S. Miller.

Esta certificación sólo testifica la autenticidad de la firma del funcionario que firmó el documento adjunto, la capacidad en la cual actuó dicho funcionario, y cuando así se requiere, la identidad del sello o timbre que lleva el documento. Esta certificación no implica que el contenido del documento sea correcto ni que esté aprobado por el Estado de Michigan.

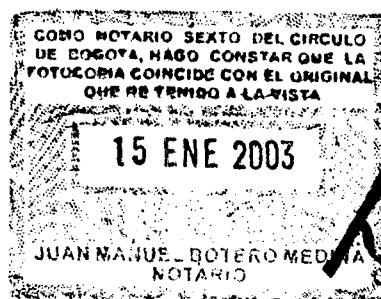
Es traducción fiel y completa de la cual se deja copia en esta oficina para su confrontación.

COLO 13518-803-001-3

WPCL 002F - ID 2152

ENE 10/03

Maria E. Martelo
TRADUCCION No: 07-01103
MARIA ELVIRA MARTELO
Traductora e Interprete Oficial
Inglés - Español - Inglés
Res. No. 2879 Min. Justicia



NO SE ASUME
RESPONSABILIDAD DEL TEXTO

2003 JAN 14 A 11:35

Jefe de LEGALIZACIONES
SANTAFE DE BOGOTA D.C.

MINISTERIO DE RELACIONES
EXTERIORES

002200

CUYA FIRMA APARECE EN EL
DOCUMENTO DESEMPEÑA
LAS FUNCIONES INDICADAS

10

COMO NOTARIO SEXTO DEL CIRCULO DE SANTAFE DE BOGOTA, HAGO CONSTAR QUE LA(S) ANTERIORES) FIRMA(S) PUESTA(S) EN EL PRESENTE DOCUMENTO CON C.S. No. (S)

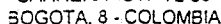
ES(S)O(S) AUTENTICA(S)

SANTAFE DE BOGOTA, 14 JAN 2003

COMO NOTARIO SEXTO DEL CIRCULO DE BOGOTA, HAGO CONSTAR QUE LA FOTOCOPIA CONCIERNE CON EL ORIGINAL QUE ME TENGO A LA VISTA.

15 ENE 2003

JUAN MANUEL BOTERO MEDINA
NOTARIO



Representation and Power-of-Attorney with notarial authentication and Apostille for patents, utility models, industrial designs, trademarks, commercial names, trade emblems, domain names, slogans, licenses.

REPRESENTATION AND POWER-OF-ATTORNEY

DOW GLOBAL TECHNOLOGIES INC.

domiciliado (s) en/of 1790 Building, Washington Street

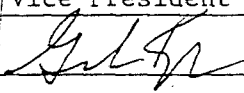
Midland, Michigan 48676, U.S.A.

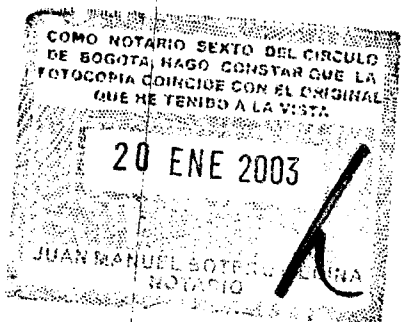
Además, confiero (conferimos) poder a los abogados arriba nombrados, para obtener la protección de mi (nuestra) propiedad industrial y contra la competencia desleal, a cuyo efecto autorizo (amos) a los dichos apoderados para que me (nos) representen ante cualesquiera entidades o personas, ejerzan o no jurisdicción, especialmente ante las del orden administrativo, contencioso- administrativo y judicial, así como los tribunales de arbitramento, en todo lo concerniente a los siguientes asuntos: a) Obtención de patentes de invención, modelos de utilidad y diseños industriales; el registro de marcas de fábrica, colectivas, defensivas, de servicio, lemas comerciales, nombres comerciales, nombres de dominio, variedades vegetales y denominaciones de origen; su renovación, traspaso, cambio de nombre o domicilio, cancelación voluntaria; y el registro de licencias de uso de esos derechos; b. Las acciones de competencia desleal, protección del consumidor, peticiones u observaciones, cancelación administrativa y judicial, cautela, medidas cautelares, concesión de licencias, la tutela, la protección de secretos industriales, y en general los juicios civiles, penales, administrativos o contencioso administrativos relacionados con

in due compliance with the terms of paragraph 1st. of article 543 of the Commercial Code, do hereby appoint EMILIO FERRERO, tpa. 31929; GONZALO PERDOMO, tpa. 831; LUZ CLEMENCIA DE PAEZ, tpa. 23555 and GERMAN CAVELIER, tpa. 832, domiciled at Carrera 4a N° 72 35, Bogotá, Colombia, the first as principal and the others as alternates, in the order of their appointment, as our representatives for all industrial property matters with faculty to receive notifications and to appoint attorneys in and out of Court. The first alternate shall replace the principal in case of his absence or of temporal or definitive impediment. The same rule will apply when the second alternate representative is to replace the first one, or when the third is to replace the first or second alternates in their turn, or both of them, second. In such cases, the principal representative, or the first or second alternates in their turn, or both of them, shall state their intention not to act as representatives through a declaration given before a Notary Public in Colombia, or before a Notary or another Public Official or a Colombian Consul abroad; and in case of impediment, the appropriate certificate shall suffice. If any of the appointees were to be definitely absent, the following alternate shall take his place. When the absence or impediment of the principal representative, or of the first or second alternate in their turn ceases, the representation can be taken again in their sequence, by the principal, the first or second alternate depending on case, and the mere fact or its exercise will suffice to exercise the representation by the first, second or third alternates in their turn, shall end at such time. In addition, we grant a Power of Attorney in favor of the above named attorneys at law so that they may obtain the protection of my (our) industrial property and against the unfair competition for which purpose I (we) authorize the said attorneys to represent me (us) before any authorities or persons, with or without jurisdiction, specially those administrative, administrative contentious and judicial, as well as before Arbitration Court, in all matters concerning the following: a) Obtainment of patents, utility models and industrial designs; the registration of trademarks, collective, defensive and service marks, slogans, commercial names, trade emblems, domain names, vegetal varieties and denominations of origin; its renewal, assignment, change of name or domicile, voluntary cancellation; and the registration of licenses of use of the above rights; b) Actions of unfair competition, protection to the consumer, oppositions or observations, administrative or Court cancellation, nullity, infringement, granting of licenses, the action for writ mandamus, the protection of trade secrets, and in general the civil, penal, and administrative suits relating to those rights; actions and suits

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estos derechos; acciones y procesos que promovamos o se nos promuevan. Y para que en todos los asuntos arriba determinados, puedan sustituir este mandato, transigir, conciliar, admitir los hechos del proceso, desistir, cancelar, recibir, renunciar, y ratificar los actos de agentes oficiosos.

instituted by me (us) or against me (us). And that in all cases they may substitute this Power of Attorney, compromise, concede the facts of the case, desist, cancel, receive, resign, and ratify acts done by me (us) or my (our) representatives without a power of attorney.

Dado y firmado hoy/Given and signed this _____
en/in Midland, Michigan 48674, U.S.A.
por/by GRAHAM E. TAYLOR
Vice President
Firma  Signature



cellulose aqueous solution is then pumped through the three columns in series in the down flow direction at a rate of about 0.1 gallons per minute per square foot of resin. After passing through the columns, the hydroxypropyl methylcellulose is dewatered on a drum dryer.

Results

A sample of purified and dewatered hydroxypropyl methylcellulose is collected and analyzed. The neutral two percent solution color of the purified and dewatered hydroxypropyl methylcellulose is measured as about 15 APHA units. The salt (NaCl) content of the purified and dewatered hydroxypropyl methylcellulose is not detectable (less than 0.01 percent). The powder whiteness value of the purified and dewatered hydroxypropyl methylcellulose is measured as about 65 units.

What is claimed is:

1. A method of removing color bodies from a water-soluble polysaccharide ether containing such color bodies comprising:

(a) contacting an aqueous solution which comprises a water-soluble polysaccharide ether containing color bodies, with an anion exchange resin in an amount and at conditions effective to associate the color bodies with the resin; and

(b) separating the resin from the polysaccharide ether so as to result in the polysaccharide ether having a reduced concentration of color bodies;

wherein the water-soluble polysaccharide ether is in the aqueous solution in a concentration greater than about 1 weight percent.

2. The method of claim 1 wherein the anion exchange resin is derived from the group consisting of an epoxy resin polymer, an acrylic based copolymer, and a copolymer composition of styrene-divinylbenzene.

3. The method in claim 2 wherein the epoxy resin polymer is an epoxy-amine copolymer.

4. The method in claim 2 wherein the acrylic based copolymer is an acrylic-divinylbenzene copolymer.

5. The method in claim 1 wherein the anion exchange resin is a weak-base resin.

6. The method in claim 1 wherein the anion exchange resin is a strong-base resin.

7. The method in claim 1 wherein the anion exchange resin is a macroporous resin.

8. The method in claim 1 wherein the anion exchange resin is a gel-type resin.

9. The method in claim 1 wherein the resin is a mixed bed of resins comprising a strong-base styrene-divinylbenzene gel-type resin and a strong-acid cation exchange styrene-divinylbenzene resin.

10. The method in claim 1 wherein the resin is an absorptive, porous, post-crosslinked resin.

11. The method in claim 1 wherein the water-soluble polysaccharide ether is derived from a natural gum.

12. The method in claim 11 wherein the natural gum is selected from the group consisting of arabic, xanthan and guar.

13. The method in claim 1 wherein the water-soluble polysaccharide ether is derived from a dextran.

14. The method in claim 1 wherein the water-soluble polysaccharide ether is derived from a starch.

15. The method of claim 1 wherein the water-soluble polysaccharide ether is a water-soluble cellulose ether.

16. The method of claim 15 wherein the water-soluble cellulose ether is selected from the group consisting of alkylcellulose, hydroxyalkyl alkylcellulose, methylcellulose, hydroxypropyl methylcellulose, hydroxyethyl methylcellulose, ethyl methylcellulose, hydroxyethyl hydroxypropyl methylcellulose, hydroxyethylcellulose, and hydroxypropylcellulose.

17. The method of claim 1 wherein the polysaccharide ether is present in the aqueous solution in a concentration greater than about 5 weight percent.

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US005218107A

United States Patent [19][11] **Patent Number:** **5,218,107****Schulz**[45] **Date of Patent:** **Jun. 8, 1993**

[54] **REMOVAL OF UNDESIRABLE MATERIAL
FROM WATER-SOLUBLE CELLULOSE
ETHER AQUEOUS SOLUTIONS**

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[21] **Appl. No.:** **814,525**

[22] **Filed:** **Dec. 30, 1991**

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C08B 11/20; C08B 11/193**

[52] **U.S. Cl.** **536/84; 536/85;
536/90; 536/91; 536/95; 536/96; 536/97;
536/98; 536/99; 536/100**

[58] **Field of Search** **536/84, 85, 90, 91,
536/95, 96, 97, 98, 99, 100**

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Primary Examiner—Nathan M. Nutter

[57] **ABSTRACT**

This invention is a process of removing water-soluble impurities from a water-soluble cellulose ether aqueous solution containing such water-soluble impurities comprising treating an aqueous solution comprising a water-soluble cellulose ether and a water-soluble impurity by means of ultrafiltration and diafiltration at conditions effective to produce a treated aqueous solution having a reduced concentration of the water-soluble impurity and an increased concentration of the water-soluble cellulose ether.

14 Claims, No Drawings

10-1
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The brine solution is neutralized with concentrated hydrochloric acid inasmuch as the membrane cartridge cannot tolerate a pH above 9 or below 4. The brine solution is then pumped into a 300 gallon (1.1 m³) feed tank. The brine solution is filtered through a 25 × 10⁻⁶ m cartridge filter as it is transferred from the drums.

The brine solution is fed through the membrane vessel tube to concentrate the water-soluble cellulose ether from 1.6 weight percent to 4.0 weight percent HPMC at a 10.7 weight percent sodium chloride concentration.

Cellulose ether concentrations are determined by allowing the cellulose ether to react with phenol and sulfuric acid and subsequently performing a colorimetric determination. All cellulose ether concentrations are reported by this technique. However, all reported cellulose ether concentrations have a ±20 percent error factor due to impurities interfering with the concentration analysis. Sodium chloride levels are determined by titration with silver nitrate.

The HPMC waste brine is recirculated at 3.3 gallons per minute (1.3 × 10⁻² m³ per minute) with a pressure drop of 100 to 600 psi (7 × 10⁵ to 4 × 10⁶ Pa), but more typically at 300 psi (2 × 10⁶ Pa), across the membrane cartridge and at a temperature of 20° to 45° C. Permeate flux (the rate at which brine passes through the membrane based on 25° C.) is started at 10 gal/day/ft² (0.4 m³/day/m²) and decreases to 6.4 gal/day/ft² (0.3 m³/day/m²).

Water, 700 pounds (318 kg), is then added to a recirculating loop in order to wash out salt and other water-soluble impurities by diafiltration. During diafiltration, the water addition rate is set equal to the permeate rate so that diafiltration is done independently of concentration of the HPMC. Diafiltration is continued until the sodium chloride level is reduced to 4.1 weight percent of the brine solution, at which time the permeate flux has increased to 11.9 gal/day/ft² (0.5 m³/day/m²).

Concentration of the HPMC is then resumed to concentrate the brine solution to 11.4 weight percent HPMC, at which time the permeate flux has decreased to 5.0 gal/day/ft² (0.2 m³/day/m²).

Diafiltration is then continued for an additional 1,134 pounds (514 kg) water. This reduces the sodium chloride level to 0.4 weight percent of the brine solution, at which time the permeate flux has increased to 7.1 gal/day/ft² (0.3 m³/day/m²).

Concentration of the HPMC is then resumed until the HPMC concentration reaches 15.2 weight percent HPMC of the brine solution, at which time the permeate flux has decreased to 3.7 gal/day/ft² (0.15 m³/day/m²).

Diafiltration is resumed again using 224 pounds (102 kg) water to reduce the sodium chloride level to 0.07 weight percent of the brine solution, at which time the permeate flux has increased to 10 gal/day/ft² (0.4 m³/day/m²).

Concentration of the HPMC is again resumed until the HPMC concentration reaches 19.6 weight percent of the brine solution, at which time the permeate flux has decreased to 2.4 gal/day/ft² (0.1 m³/day/m²). A portion of this concentrate is dried to yield a solid HPMC product with 0.7 weight percent salt. Another portion of the concentrate is recovered by adding about 5 g of the concentrate to about 22.2 g of wet (55 weight percent HPMC) METHOCEL™ K35 hydroxypropyl methylcellulose washed filter cake. The concentrate and washed filter cake are mixed and dried, representing a recovered yield.

Total cellulose can be determined by drying samples to determine total solids and correcting for salt. Infrared and carbon-13 nuclear magnetic resonance spectroscopy (NMR) and size exclusion chromatography show the contents of the concentrate to be cellulose with no significant residues other than salt.

The process described above is then used on the following HPMC waste brine solutions:

a. A waste brine solution wherein the product HPMC from which the waste brine is taken has a viscosity of about 4,000 centipoise (4,000 mPa.s) as a 2 weight percent aqueous solution at 20° C. (available from The Dow Chemical Company as METHOCEL™ K4M hydroxypropyl methylcellulose).

b. A waste brine solution wherein the product HPMC from which the waste brine is taken has a viscosity of about 4,000 centipoise (4,000 mPa.s) as a 2 weight percent aqueous solution at 20° C. (available from The Dow Chemical Company as METHOCEL™ E4M hydroxypropyl methylcellulose).

c. A waste brine solution wherein the product HPMC from which the waste brine is taken has a viscosity of about 4,000 centipoise (4,000 mPa.s) as a 2 weight percent aqueous solution at 20° C. (available from The Dow Chemical Company as METHOCEL™ F4M hydroxypropyl methylcellulose).

Several properties of the recovered water-soluble cellulose ethers are shown in Table I. The substitutions of the recovered material are close to the specifications of the parent products. The actual substitution of the parent products is not known, however, so it is not conclusive whether any differences are due to the recovered low molecular weight fraction or due to the parent material.

TABLE I

Parent Brine Type	Analysis of Recovered Cellulose Ether			
	2 Percent Viscosity (cps (mPa · s))	Percent NaCl	Percent Methoxyl Substitution	Percent Propoxyl Substitution
K35	1.70 (28-42)	.03 (5.0)	22.01 (19-24)	10.76 (7-12)
K4M	2.02 (3,500-5,600)	.06 (2.0)	21.89 (19-24)	10.97 (7-12)
E4M	1.64 (3,500-5,600)	3.29 (2.0)	24.38 (28-30)	13.05 (7-12)
F4M	1.44 (3,500-5,600)	1.27 (1.5)	25.50 (27-30)	6.26 (4-7.5)

() Indicates Specification Range or Maximum for Parent Product

Testing of the recovered water-soluble cellulose ethers in tape joint compounds, ceramic tile mortar, and paint showed no significant performance differences from the parent water-soluble cellulose ether products.

What is claimed is:

1. A process of removing a water-soluble purity from a water-soluble cellulose ether aqueous solution containing such water-soluble impurity comprising treating an aqueous solution comprising a thermally-gelling, water-soluble cellulose ether and an alkali metal salt impurity;

a. by means of ultrafiltration wherein the permeate flux decreases during the use of the means of ultrafiltration; and

b. by means of diafiltration wherein the permeate flux increases during the use of the means of diafiltration

at conditions effective to produce a treated aqueous solution having a reduced concentration of the alkali



US005840882A

United States Patent [19]**Doenges et al.**[11] **Patent Number:** **5,840,882**[45] **Date of Patent:** **Nov. 24, 1998**[54] **PROCESS FOR PREPARING CELLULOSE
ETHERS WITH RECOVERY OF ALKALI**[75] Inventors: **Reinhard Doenges**, Bad Soden; **Rudolf
Ehrler**, Floersheim, both of Germany[73] Assignee: **Clariant GmbH**, Frankfurt, Germany[21] Appl. No.: **911,946**[22] Filed: **Aug. 20, 1997**[30] **Foreign Application Priority Data**

Aug. 22, 1996 [DE] Germany 196 33 826.3

[51] **Int. Cl.⁶** **C07H 1/00**; C07H 15/08[52] **U.S. Cl.** **536/91**; 536/85; 536/124[58] **Field of Search** 536/85, 91, 124[56] **References Cited****U.S. PATENT DOCUMENTS**

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Primary Examiner—Gary L. Kunz[57] **ABSTRACT**

Cellulose ethers which contain hydroxyalkyl groups are prepared, with the recovery of sodium hydroxide, by cellulose being etherified with alkylene oxide in a water-containing organic suspension medium in the presence of at least 1.0 mol of sodium hydroxide per mole of anhydroglucose units, the water-containing organic suspension medium being separated off after the etherification reaction is complete, the crude cellulose ether being extracted with a suitable solvent mixture, the lower-boiling organic components being distilled off from the solvent mixture after the extraction, the water-containing organic suspension medium separated off being added to the distillation residue of the solvent mixture to form a multiphase mixture, the bottom phase of the multiphase mixture being separated and the sodium hydroxide present in the bottom phase being reused.

15 Claims, 1 Drawing Sheet(5)
98

Example 1

Preparation of hydroxyethylcellulose (HEC)

In a 2 l glass reactor, 85.0 g of linters pulp were suspended in 800 g of t-butanol/methanol/water in a weight ratio of 87/1/12. This was inerted three times, and 56 g of 50% strength sodium hydroxide solution were then added. After stirring for 45 min at room temperature, 125 ml of ethylene oxide were added, followed by heating to 40° C. for 1 h and to 80° C. for a further 1.5 h. The mixture was then cooled to less than 30° C.

Separation of the suspension medium phase

After the reaction batch had been cooled it was transferred, without neutralization, to a glass filter nutsche pressurized with nitrogen. The batch was filtered off with suction. The suspension medium phase mainly comprised t-butanol, glycols and a small amount of sodium hydroxide solution. The suspension medium phase was collected in a separate vessel.

Washing of crude HEC

The filter cake was suspended in 1 l of a solvent mixture of t-butanol/methanol/water 65/30/5 and again transferred to the nutsche. The filter cake was further eluted with the same solvent mixture. Initially the conductivity of the eluate was about 1300 μ S and then rose to 3500 μ S. After 2440 g of solvent mixture had passed through, the conductivity of the final eluate was 780 μ S. In total, about 16 kg of solvent were consumed per kg of HEC.

Neutralization

The HEC was slurried once more in the same solvent mixture and neutralized by concentrated hydrochloric acid being added to the suspension. Filtration took place with strong suction, and the filtrate was worked up together with the extraction medium.

Drying

The moist product (1103 g, 16.5% solid, 5.9% water) was dried at 75° C. This produced 170 g of an HEC with a solids content of 93.4% and a percentage of 5.6% of sodium chloride, based on the solid. The viscosity was 60,000 mPa.s. The molar degree of substitution (MS) was 3.1.

Distillation of the eluate

The solvent mixture from the extraction was subjected to simple distillation on a rotary evaporator (200 mbar, 65° C.). The distillate consisted of 60.4% of t-butanol, 34.0% of methanol and 5.6% of water and was therefore, having been fortified with t-butanol, suitable once more for use in the extraction.

Separation of the sodium hydroxide solution

The aqueous distillation residue (70 g) was combined with the suspension phase separated off from the etherification process, heated to 60° C. and transferred to a separator. Two defined phases formed. Between the two phases a further layer formed which was an emulsion of hemicelluloses, glycols, dissolved HEC and sodium hydroxide solution. The bottom phase was separated off. This produced 58 g of 25.6% strength sodium hydroxide solution which still contained 5.1% of glycols, 1% of methanol and 1.3% of t-butanol. The interlayer was distilled together with the upper, organic phase.

The separated sodium hydroxide solution was suitable for reuse, without further work-up. In this example, 53% of the sodium hydroxide solution used were recycled.

Distillation of the suspension medium

The separated upper phase was distilled on a rotary evaporator (200 mbar, 60° C.). The distillate (557 g) consisted of 90.8% of t-butanol, 5.4% of water and 1% of methanol (remainder: t-butyl glycol, methyl glycol) and was therefore, having been made up with t-butanol, reusable as a suspension medium.

Example 2

Use of recovered sodium hydroxide solution

This experiment was carried out as described in Example 1. The sodium hydroxide solution used, however, comprised 112 g of 25% strength sodium hydroxide solution which had been recovered by recycling from previous experiments. The molar degree of substitution (MS) of the product was 3.0, the viscosity of a 2% strength aqueous solution was 55,000 mPa.s.

Example 3

Washing-out by suspension in the extraction medium

This experiment was carried out as described in Example 1. Extraction of the crude product was effected, however, by the crude product being suspended 4 times, each time in 780 g of the solvent mixture, followed by filtration with suction in a nutsche. The conductivity of the final eluate was 580 μ S. The solvent consumption was about 20 kg/kg of HEC.

In this experiment 65% of the sodium hydroxide solution were able to be recovered.

Example 4

Preparation of hydroxyethylcellulose (HEC)

In a glass reactor, 85.0 g of spruce pulp were suspended in 800 g of t-butanol/ethanol/water in a weight ratio of 85/5/10. This was inerted three times, and 56 g of 50% strength sodium hydroxide solution were then added. After stirring for 45 min at room temperature, 110 ml of ethylene oxide were added, followed by heating to 40° C. for 1 h and to 80° C. for a further 1.5 h. The mixture was then cooled to less than 30° C.

Separation of the suspension medium phase

After the reaction batch had cooled, filtration by suction was carried out on a filter nutsche. This produced 700 g of suspension medium phase.

Washing of crude HEC

The filter cake was suspended in 1 l of a solvent mixture of t-butanol/ethanol/water in a weight ratio of 20/75/5 and again transferred to the nutsche. The filter cake was further eluted with the same solvent mixture. Initially the conductivity of the eluate was about 1200 μ S and then rose to 3200 μ S. After 2460 g of washing medium had passed through, the conductivity obtained for the last eluate was 390 μ S. The solvent consumption overall was about 16 kg/kg of HEC.

Neutralization

The HEC was slurried once more in the same solvent mixture and neutralized by acetic acid being added. Filtration took place with strong suction, and the filtrate was worked up together with the extraction medium.

Drying

The moist product was dried at 70° C. This produced 172 g of an HEC with a solids content of 97.8% and a percentage of 8.2% of sodium acetate, based on the solid. The viscosity of a 2% strength aqueous solution was 180 mPa.s. The molar degree of substitution (MS) of the product was 3.1. The product was soluble in water at room temperature to 99.5%.

Distillation of the eluate

The solvent mixture from the extraction was distilled on a rotary evaporator (200 mbar, 65° C.). The distillate consisted of 21.6% of t-butanol, 72.9% of ethanol and 5.6% of water and was therefore, having been fortified with ethanol, suitable once more for use in the extraction.

Separation of the sodium hydroxide solution

The aqueous distillation residue (43 g) was combined with the suspension medium separated off from the etherification process, heated to 60° C. and transferred to a separator. Two phases formed. The bottom phase was separated off. This produced 64 g of 23% strength sodium

hydroxide solution which still contained 1.6% of glycols and 0.4% of t-butanol. The interphase was distilled together with the upper, organic phase.

The separated sodium hydroxide solution was suitable for reuse, without further work-up. In this example, 53% of the sodium hydroxide solution used were recycled.

Distillation of the suspension medium

The separated upper phase was distilled on a rotary evaporator (200 mbar, 60° C.). The distillation (620 g) consisted of 87.9% of t-butanol, 6.4% of water and 5.7% of ethanol (remainder: t-butyl glycol, ethyl glycol) and was therefore, having been made up with ethanol, reusable as a suspension medium.

Example 5

Preparation of hydroxyethylcellulose (HEC)

In a glass reactor, 85.0 g of spruce pulp were suspended in 800 g of 85% strength aqueous isopropanol. This was inerted three times, and 56 g of 50% strength sodium hydroxide solution were then added. After stirring for 45 min at room temperature, 153 ml of ethylene oxide were added, followed by heating to 40° C. for 1 h and to 80° C. for a further 1.5 h. The mixture was then cooled to less than 30° C.

Separation of the suspension medium phase

After the reaction batch had cooled, filtration by suction was carried out on a filter nutsche.

Washing of crude HEC

The filter cake was suspended in 1 l of a solvent mixture of isopropanol/methanol/water in a weight ratio of 69/25/6 and again transferred to the nutsche. The filter cake was further eluted with the same solvent mixture. Initially the conductivity of the eluate was about 1480 μ S and then rose to 3300 μ S. After 3280 g of washing medium had passed through, the conductivity obtained for the last eluate was 540 μ S.

Neutralization

The HEC was slurried once more in the same solvent mixture and neutralized by 11.6 g of acetic acid being added. Filtration took place with strong suction, and the filtrate was worked up together with the extraction medium.

Drying

The moist product was dried at 70° C. This produced 154 g of a hydroxyethylcellulose with a solids content of 97.8% and a percentage of 10.2% of sodium acetate, based on the solid. The viscosity of a 2% strength aqueous solution was 73,000 mPa.s. The molar degree of substitution (MS) was 2.8. The product was soluble in water at room temperature to 97%.

Distillation of the eluate

3688 g of the solvent mixture from the extraction was distilled on a rotary evaporator (250 mbar, 65° C.). The distillate (3570 g) consisted of 70.2% of isopropanol, 22.6% of methanol and 7.3% of water and was suitable once more for use in the extraction.

Separation of the sodium hydroxide solution

The aqueous distillation residue (113 g) was combined with the suspension medium separated off from the etherification process, heated to 60° C. and transferred to a separator. Two phases formed. The bottom phase was separated off. This produced 67 g of 22% strength sodium hydroxide solution which still contained 1.3% of glycols and 2.5% of isopropanol.

The separated sodium hydroxide solution, having been made up with pure sodium hydroxide, was suitable for reuse, without further work-up. In this example, 57% of the sodium hydroxide solution used were recycled.

Distillation of the suspension medium

The separated upper phase was distilled on a rotary evaporator (200 mbar, 60° C.). The distillate (794 g), having a composition of 85.8% of isopropanol, 13.4% of water and 0.8% of methanol, almost matched the mixture used and was therefore reusable as a suspension medium.

What is claimed is:

1. A process for the preparation of hydroxyalkyl cellulose ethers, comprising:

- (a) etherifying cellulose with an alkylene oxide in a water-containing organic suspension medium in the presence of an effective quantity of inorganic alkali which is approximately at least one equivalent of inorganic alkali per mole of anhydroglucose units in the cellulose, to obtain a crude hydroxyalkylcellulose ether;
- (b) separating the water-containing organic suspension medium from the crude hydroxyalkyl cellulose ether, said crude hydroxyalkyl cellulose ether containing a major amount of said effective quantity of inorganic alkali, the thus-separated water-containing organic suspension medium containing a minor amount of said effective quantity of inorganic alkali;
- (c) extracting essentially said major amount from said crude hydroxyalkyl cellulose ether, with a liquid organic extracting medium capable of extracting inorganic alkali, to obtain an eluate containing essentially said major amount and separating out, from said eluate, hydroxyalkylcellulose contaminated with essentially only a residual amount of inorganic alkali;
- (d) treating with acid said hydroxyalkylcellulose contaminated with only a residual amount of inorganic alkali to neutralize essentially only said residual amount of inorganic alkali;
- (e) separating essentially all of said inorganic alkali in said eluate from said eluate;
- (f) recovering the thus-separated inorganic alkali in sufficient purity for use in the etherification of cellulose with an alkylene oxide or for use in partial hydrolysis of cellulose.

2. The process of claim 1, wherein

said inorganic alkali is sodium hydroxide, and said cellulose-digesting amount is at least 1.0 mole of sodium hydroxide per mole of anhydroglucose units in the cellulose which is etherified;

said extracting medium comprises an ORGANIC solvent mixture optionally containing water; and, after said extracting, said eluate is distilled to recover essentially purified organic solvent mixture and a distillation residue comprising sodium hydroxide;

said water-containing organic suspension medium is combined with said distillation residue to form a mixture having a plurality of phases including a lower phase; and

sodium hydroxide is separated from said lower phase and utilized in the preparation of a cellulose ether from cellulose.

3. The process of claim 2, wherein the thus-separated sodium hydroxide is recycled to said step (a) of the process.

4. The process of claim 2, wherein the thus-recovered, essentially purified organic solvent mixture is recycled for use in said step (c).

5. The process of claim 1, wherein said water-containing organic suspension medium contains a monohydric alcohol having from 2 to 4 carbon atoms.



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DEUTSCHLAND



DEUTSCHES
PATENTAMT

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51 Int. Cl.⁵:
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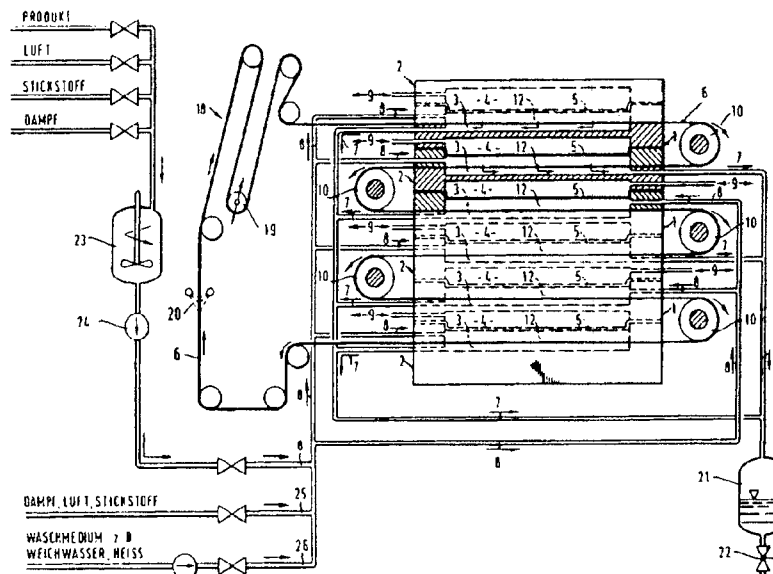
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- 54 Verfahren zum Abtrennen und Reinigen von Celluloseethern aus einer Celluloseethersuspension
57 Verfahren zur Abtrennung von Celluloseethern aus einer Celluloseethersuspension und Reinigung der abgetrennten Celluloseether in dem man die Abtrennung und Reinigung mit Hilfe eines taktweise arbeitenden Druckpreßfilters vornimmt.



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Wasser und durch die Membranpressung wird dann die Druckhaltung überwunden und die Mutterlauge bzw. die Waschflüssigkeit aus dem Kuchen entfernt. Je nach Druckvorgabe der Druckhaltung lassen sich bei kommerziell zu erhaltenden Geräten Temperaturen bis zu 200°C erzielen.

Da die Flocktemperatur von Celluloseethern außer von den oben aufgeführten Substitutionskriterien auch vom Elektrolytgehalt des umgebenden wäßrigen Mediums in der Weise abhängt, daß die Flocktemperatur mit steigender Elektrolytkonzentration sinkt, ist es häufig der Fall, daß die Abtrennung der salzhaltigen Mutterlauge aus der Reaktion der Celluloseetherherstellung bei Normaldruck und Temperaturen unter 100°C durchgeführt werden kann, während die nachfolgenden Reinigungsschritte bei höheren Temperaturen und unter Druck durchgeführt werden müssen.

Ist der Filterkuchen (12) ausreichend gereinigt, so kann gegebenenfalls durch den Kanal (8) Druckluft eingepreßt werden, um den Filterkuchen (12) zu trocknen. Anschließend wird die Schließvorrichtung für die Platten (1, 2), die die Filterkammern bilden, gelöst und durch eine Weiterbewegung des Filterkuchens (6) wird der Filterkuchen auf die Umlenkorgane (10) geführt und dort (11) abgestreift. Fig. 3 zeigt eine Filterkammer in geöffnetem Zustand. Die zum Öffnen erforderliche Filtertuchlänge wird einem Reservoir (18) mit Tänzerwalze (19) entnommen. Durch die gegenläufige Bewegung des Filterkuchens (6) erfolgt der Austrag des Filterkuchens (12) von Filterkammer zu Filterkammer abwechselnd nach links oder rechts.

Die folgenden Ausführungsbeispiele verdeutlichen das durch den Einsatz eines Druckpreßfilters gekennzeichnete Verfahren zum Abtrennen und Reinigen von Celluloseethern, ohne daß die Erfindung auf diese Beispiele beschränkt ist.

Es wird deutlich, daß auch bei Celluloseethern, die aufgrund ihrer niedrigen Viskosität bzw. ihres hohen Flockpunktes nach anderen Verfahren nur schwer zu reinigen sind, durch den Einsatz von wenig Waschflüssigkeit hervorragende Reinigungseffekte zu erzielen sind.

Beispiel 1

Nach bekanntem Verfahren wurde eine Methylhydroxypropylcellulose — MHPC — hergestellt, die nach Entfernung des für die Reaktion verwendeten Suspensionsmittels folgende Werte aufwies:

Wasserfeuchte 31%
Kochsalzgehalt 38%

Das salzhaltige Produkt wurde zunächst in 95 bis 100°C heißem Wasser suspendiert (Aufschlagverhältnis 2,7 : 1 bezogen auf Rohware) und in einem unter Druck zu betreibenden Behälter (23) bevorratet. Die Beschickung des taktweise arbeitenden Druckpreßfilters erfolgt mittels einer Pumpe (24). Das Filtertuch des Druckpreßfilters bestand aus Polypropylen-Monofilament mit einer Luftdurchlässigkeit von 2 m³/m² min bei 1500 Pa.

Am Druckpreßfilter lief folgender Zyklus ab:

1. Konditionieren der Filterkammern mit Dampf (Zuleitung 25) für 30 s
2. Befüllen der Filterkammern mit Celluloseethersuspension

3. Abpressen des Filterkuchens mit der Membran für 3 min bei 16 bar
4. Konditionieren der Filterkammern mit Dampf für 30 s
5. Zugabe von Waschwasser (Zuleitung 26) bei 98°C im Verhältnis 0,55 : 1 bezogen auf Rohware
6. Abpressen des Filterkuchens mit der Membran für 3 min bei 16 bar
7. Öffnen der Filterkammern und Austragen des Filterkuchens (12)

Für das bei (7) ausgetragene Produkt wurde eine Restfeuchte von 48% bestimmt. Der Restkochsalzgehalt betrug 0,4%. Die MHPC wies folgende Produktmerkmale auf:

Höppler Viskosität (20°C, 1,9%ige Lösung) 150 mPa · s

Substitutionsgrad

- a) Methoxyl 25,8%
- b) Hydroxypropyl 8,5%

Der Kochsalzgehalt kann durch Wahl der Waschwassermenge, die Restfeuchte durch Wahl des Abpreßdruckes in weiten Grenzen einreguliert werden.

Beispiel 2

Nach bekannten Verfahren wurde eine Methylhydroxyethylcellulose — MHEC — hergestellt, die nach Entfernung des für die Reaktion verwendeten Suspensionsmittels folgende Werte aufwies:

Wasserfeuchte 33%
Kochsalzgehalt 36%

Das salzhaltige Produkt wurde zunächst in 95 bis 100°C heißem Wasser suspendiert (Aufschlagverhältnis 3,1 : 1 bezogen auf Rohware) und in einem unter Druck zu betreibenden Behälter bevorratet. Die Beschickung des taktweise arbeitenden Druckpreßfilters erfolgt wie in Beispiel 1. Es wurde das gleiche Filtertuch verwendet.

Am Druckpreßfilter lief folgender Zyklus ab:

1. Konditionieren der Filterkammer mit Dampf für 30 s
2. Befüllen der Filterkammern mit Celluloseethersuspension
3. Abpressen des Filterkuchens mit der Membran für 3 min bei 16 bar
4. Konditionieren der Filterkammer mit Dampf für 30 s
5. Zugabe von Waschwasser bei 98°C im Verhältnis 2,25 : 1 bezogen auf Rohware
6. Abpressen des Filterkuchens mit der Membran für 3 min bei 16 bar
7. Öffnen der Filterkammern und Austragen des Filterkuchens

Für das ausgetragene Produkt wurde eine Restfeuchte von 39% bestimmt. Nach Trocknen des Produkts wurde ein Restkochsalzgehalt von < 0,1% ermittelt. Die MHEC wies folgende Produktmerkmale auf:

Höppler Viskosität (20°C, 1,9%ige Lösung) 24 600 mPa · s

Substitutionsgrad

- a) Methoxyl 29%
- b) Hydroxyethyl 2,6%

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Beispiel 3

Nach bekanntem Verfahren wurde eine Methylhydroxyethylcellulose — MHEC — hergestellt, die nach Entfernung des für die Reaktion verwendeten Suspensionsmittels folgende Werte aufwies:

Wasserfeuchte 40%
Kochsalzgehalt 27%

Das salzhaltige Produkt wurde zunächst in 110°C heißem Wasser suspendiert (Aufschlagverhältnis 2,9 : 1 bezogen auf Rohware) und in einem unter Druck von 1,4 bar zu betreibenden Behälter bevorratet. Die Beschickung des taktweise arbeitenden Druckpreßfilters erfolgt wie bei Beispiel 1 (Filtertuch wie bei Beispiel 1).

Am Druckpreßfilter lief folgender Zyklus ab:

1. Konditionieren der Filterkammern mit Dampf für 30 s
2. Befüllen der Filterkammern mit Celluloseethersuspension
3. Abpressen des Filterkuchens mit der Membran für 3 min bei 16 bar
4. Konditionieren der Filterkammern mit Dampf für 30 s
5. Zugabe von Waschwasser bei 120°C und 2 bar im Verhältnis 0,9 : 1 bezogen auf Rohware
6. Abpressen des Filterkuchens mit der Membran für 3 min bei 16 bar
7. Öffnen der Filterkammern und Austragen des Filterkuchens

Für das ausgetragene Produkt wurde eine Restfeuchte von 50% bestimmt. Der Restkochsalzgehalt betrug 0,3%. Die MHEC wies folgende Produktmerkmale auf: Höppler Viskosität (20°C, 1,0%ige Lösung) 13 000 mPa · s

Substitutionsgrad
a) Methoxyl 23,1%
b) Hydroxyethyl 9,6%

Beispiel 4

Nach bekanntem Verfahren wurde eine Natriumcarboxymethylcellulose — CMC — hergestellt. Die nach der Reaktion und Zugabe von Wasser erhaltene Suspension hatte folgende Zusammensetzung: CMC 7%, Kochsalz 2%, Isopropanol 52%, Wasser 38% und wurde direkt in das taktweise arbeitende Druckpreßfilter gepumpt. Es lief folgender Zyklus ab:

1. Befüllen der Filterkammern mit Celluloseethersuspension
2. Abpressen des Filterkuchens mit der Membran für 4 min bei 14 bar
3. Zugabe eines Isopropanol/Wasser-Gemisches, das beim Abpressen nach dem zweiten Waschschrift einer vorangegangenen Produktcharge erhalten wurde, im Verhältnis 7 : 1 (bezogen auf das Produkt)
4. Abpressen des Filterkuchens mit der Membran für 4 min bei 14 bar
5. Zugabe eines Isopropanol/Wasser-Gemisches, das beim Abpressen nach dem dritten Waschschrift einer vorangegangenen Produktcharge erhalten wurde, im Verhältnis 7 : 1

6. Abpressen des Filterkuchens mit der Membran für 4 min bei 14 bar
7. Zugabe eines Isopropanol/Wasser-Gemisches (60 : 40) im Verhältnis 7 : 1
8. Abpressen des Filterkuchens mit der Membran für 4 min bei 14 bar
9. Aufgabe von Wasserdampf für 4 min
10. Öffnen der Filterkammern und Austragen des Filterkuchens

Für das ausgetragene Produkt wurde eine Restfeuchte von 41% bestimmt. Der Restkochsalzgehalt betrug 0,4%.

Die CMC wies folgende Merkmale auf:

Höppler Viskosität (20°C, 2%ige Lösung) 26 mPa · s
Substitutionsgrad 0,76

Patentansprüche

1. Verfahren zur Abtrennung von Celluloseethern aus einer Celluloseethersuspension und Reinigung der abgetrennten Celluloseether, dadurch gekennzeichnet, daß die Abtrennung und Reinigung mit Hilfe eines taktweise arbeitenden Druckpreßfilters vorgenommen wird.
2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Abtrennung aus einer wäßrigen oder einer ein organisches Lösungsmittel enthaltenden Suspension erfolgt.
3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Reinigung mit Hilfe einer wäßrigen oder einer organischen Lösemittel enthaltenden Waschflüssigkeit vorgenommen wird, wobei sich die Lösemittel von denen in der im vorangegangenen Schritt getrennten Suspension unterscheiden können.
4. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Reinigung mehrstufig vorgenommen wird.
5. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß bei der Reinigung eine aus einem vorangegangenen Reinigungsschritt einer anderen Produktcharge erhaltene mit Salzen bzw. Nebenprodukten beladene, jedoch nicht gesättigte Flüssigkeit eingesetzt wird.
6. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Filterkuchen jeweils nach den Abtrenn- bzw. Reinigungsschritten mit Dampf beaufschlagt wird.
7. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Abtrennung bzw. Reinigung bei einer Temperatur zwischen 10 und 150°C, bevorzugt zwischen 60 und 120°C erfolgt.
8. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Abtrennung bzw. die Reinigung unter Druck durchgeführt wird.
9. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Filterkuchen durch Aufgabe von Luft oder Stickstoff entfeuchtet wird.
10. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß ein aus mehreren Kammern bestehendes Druckpreßfilter eingesetzt wird, bei dem sich am Band die Filtrationsrichtung bei der jeweils folgenden Beladung mit der nächsten Produktcharge umkehrt.

Hierzu 3 Seite(n) Zeichnungen

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[54] PURIFICATION OF CELLULOSE AND STARCH ETHERS WITH COUNTER CURRENT WASHING

[75] Inventors: Claus-Rüdiger Bernert, Walsrode; Volker Hartwig, Fallingbostel; Christian Kords, Walsrode; Detmar Redeker, Cologne, all of Fed. Rep. of Germany

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[21] Appl. No.: 323,576

[22] Filed: Nov. 23, 1980

[30] Foreign Application Priority Data

Nov. 27, 1980 [DE] Fed. Rep. of Germany 3044696

[51] Int. Cl.³ C08B 11/20; C08B 31/08

[52] U.S. Cl. 536/85; 536/96; 536/98; 536/99; 536/111

[58] Field of Search 536/85, 111

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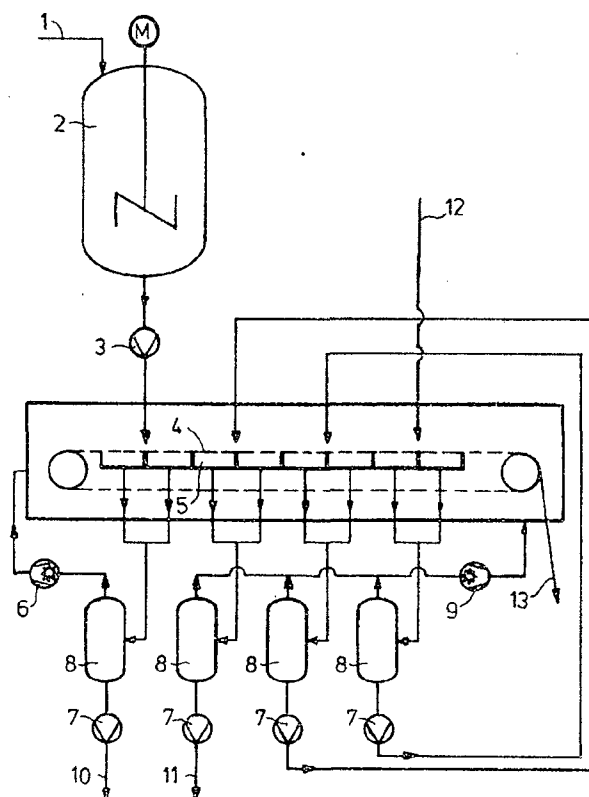
2017454 10/1971 Fed. Rep. of Germany .

Primary Examiner—A. Lionel Clingman
Attorney, Agent, or Firm—Connolly and Hutz

[57] ABSTRACT

A process for the purification and isolation of cellulose or starch ethers wherein directly after the etherification step the suspension agent and the extraction of the ether product with the purifying agent is carried out on the same continuously working filter.

6 Claims, 1 Drawing Figure



sufficient on the continuously working filter for separation of the ether and its purification.

The solids concentration of the suspension obtained after etherification may be varied within wide ranges limited by the need for fluidity and by economic considerations. A solids concentration of from 5% to 20% is preferred.

After the suspension agent has been filtered off, the ether cake is preferably dried under suction to a residual moisture content of from 40 to 70% by weight and extracted and purified in countercurrent in the following washing zone by delivery of the purification agent to the filter. The washing agent may be delivered to the ether on the filter in one or more zones, whereby the filtrate drawn through is re-used in another zone of the filter. Extraction is carried out in countercurrent at least once and preferably three times.

The washing zone is followed by a suction-drying zone into which steam or hot nitrogen may also be introduced. The ether is then separated off from the filter and further processed.

Suitable filters are those whose surface can be clearly divided into individual zones, for example plate filters, vacuum drum, pressure drum and band filters of suitable size. Drum filters have an advantage over plate filters in that they may be divided into zones of equal length. Band filters are preferably used for the process according to the invention.

The process according to the invention is particularly advantageous by virtue of its low consumption of purification agent. By virtue of the countercurrent principle, the ratio of cellulose or starch ether to washing agent may be less than 1:10. Even with a ratio of 1:7, it is still possible to obtain very good purification effects.

FIG. 1 illustrates the arrangement of a band filter. From the stirrer-equipped vessel (2), the suspension (1) of the crude ether is pumped onto the band filter (pump 3) and treated with the purifying agent (12). The filtrate passes through the circulating filter band (4) into one of the suction cells (5) which communicate with the vacuum pumps (6,9) through the filtrate-separators (8). The filtrate (11) is returned by the filtrate pumps (7) to the next washing zone and, after passing through all the zones, is worked up. The suspension medium (10) is drained off. The purified ether (13) is worked up in known manner.

EXAMPLE 1

A suspension, originating from the etherification stage, of 10% by weight of carboxymethyl cellulose in a mixture of 88% by weight of isopropanol and water is delivered through a suction cell to a vacuum drum filter 500 mm long and 700 mm in diameter at a rate of 1900 l/h. The surface of the drum is divided into twenty zones. The filtrates can be separately removed through a control head. The rotational speed amounts to approximately 1 min⁻¹. After separation of the mother liquor and drying under suction, the product is subjected to countercurrent extraction in two zones with 1600 l/h of a mixture of 65% of methanol and 35% of water introduced through nozzles. The washing medium which now contains salts is separately run off and the solvent circuit is closed by regeneration.

The remaining filter zones serve as a suction drying zone. The 20 mm thick cake is stripped from the filter by means of a knife. The purified carboxymethyl cellulose still has a moisture content of 61.5% and a sodium

chloride content of 0.45% by weight. It may be directly dried.

EXAMPLE 2

As described in Example 1, a neutralised suspension of 10% by weight of hydroxyethyl cellulose in a mixture, emanating from the etherification step, of 90% of isopropanol and 10% of water is freed from the suspension medium in a vacuum drum filter at a rate of 1900 l/h and, after drying under suction, is washed in countercurrent in two washing zones with 1500 l/h of a mixture of 30% of isopropanol, 35% of methanol and 30% of water. The product, which has a residual moisture content of 65%, contains 0.12% by weight of sodium acetate.

EXAMPLE 3

1000 l/h of a carboxymethyl cellulose suspension having the same composition as described in Example 1 are delivered to a band filter 3200 mm long and 250 mm wide. Arranged below the filter band are 8 suction cells with which a total of 2 vacuum pumps and 4 filtrate pumps are associated. An approximately 700 mm long suction-drying zone is followed by 2 or 3 extraction zones, as set forth in Table 1, to which 700-1000 l/h of a 65% methanol are delivered in countercurrent. The speed of travel of the filter band is between 16 and 38 mm/s.

The following results were obtained at the end of the suction-drying zone:

TABLE 1

Extraction Zones	2	2	3	3	3
Purification agent (l/h)	1000	700	1000	850	700
Ether thickness (mm)	16	18	20	17	18
Ether moisture (% by weight)	58	62	65	60	61
NaCl-content of the ether-cake (% by weight)	0.23	0.55	0.10	0.17	0.29

EXAMPLE 4

The procedure is as described in Example 3 using 1000 l/h of the water-diluted distillate of the spent washing agent which consists of a mixture of 22% by weight of isopropanol, 43% by weight of methanol and 35% by weight of water. The resulting NaCl-content of the cake amounts to 0.12% by weight.

EXAMPLE 5

530 l/h of a suspension of 18% by weight of carboxymethyl starch in a mixture of 89% by weight of isopropanol and 11% by weight of water are delivered to the band filter described in Example 3. Washing in three washing zones with 760 l/h of a mixture of 25% by weight of isopropanol, 45% by weight of methanol and 30% by weight of water gives a purified product containing 0.33% by weight of residual NaCl.

EXAMPLE 6

The procedure is as described in Example 3 using 850 l/h of a mixture of 68% by weight of ethanol and 32% by weight of water delivered in countercurrent to 3 washing zones. The resulting NaCl-content amounts to 0.29% by weight.

We claim:

1. A process for the continuous purification and isolation of a cellulose or starch ether suspension which comprises filtering off the suspension agent, extracting the reaction product with a purifying agent different

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SUPERINDUSTRIA Y COMERCIO

Radicacion : 00029095 00000006 Folios: 9
Fecha (AMD): 2005-08-29 17:01:42
Tramite : 002 PATENTES D 1 REGISTRO/ 444 REPUESTA
Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

Señora Jefe de la
DIVISION DE NUEVAS CREACIONES
Superintendencia de Industria y Comercio
E. S. D.

Trámite : 002
Evento : 001
Actuación: 444

Referencia : Expediente No. 00.029.095
Solicitante : DOW GLOBAL TECHNOLOGIES, INC.
Asunto : Solicitud de patente para "CÁPSULAS DE
ÉTER DE CELULOSA LIBRES DE TURBIEDAD Y
PROCESO PARA HACER LAS MISMAS"
Fecha de solicitud : 19 de abril de 2000.

1. Yo, Emilio FERRERO, obrando como apoderado del solicitante en el asunto antes identificado, me refiero al oficio número 12914, notificado por fijación en lista del 02 de junio de 2005.

2. Mediante el oficio número 12914 se puso en conocimiento del solicitante el informe que contiene las observaciones hechas por su Despacho sobre el examen de fondo de la presente solicitud de patente, las cuales resumo a continuación:

2.1. Su Despacho considera que las reivindicaciones 1 a 9 no son novedosas con relación a los documentos US 4.988.807, US 5.218.107 y US 5.840.882, porque las composiciones de la presente solicitud son iguales a las de los documentos citados.

2.2. Su Despacho establece que las reivindicaciones 10 a 20 de la solicitud carecen de nivel inventivo porque los documentos DE 4112849 y US 4.404.370 demuestran que la purificación de los éteres de celulosa ya es posible con métodos convencionales conocidos en el estado de la técnica.

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3. Atiendo a las observaciones de la manera siguiente:

3.1. Novedad. Con el objetivo de subsanar la falta de novedad de las reivindicaciones, presento junto con este escrito un nuevo capítulo reivindicatorio en el cual se han hecho las siguientes modificaciones:

- Se han combinado las reivindicaciones 1 y 5 en una nueva reivindicación 1;
- Se han modificado las reivindicaciones 2 a 5 para que incluyan los aspectos relevantes de las reivindicaciones 2, 3 y 6 a 9 originales;
- La reivindicación 10 es ahora la reivindicación 6; y
- Las reivindicaciones originales 11 a 20 ahora se resumen en las reivindicaciones 7 a 12.

Igualmente presento el recibo de pago de \$91,000.00 por concepto de modificaciones en solicitud pendiente así como el formato de modificaciones y correcciones.

Por otra parte, si bien la modificación realizada a las reivindicaciones subsana la posible falta de novedad de las mismas, deseo resaltar algunos aspectos que las diferencian definitivamente del arte previo.

La nueva reivindicación 1 está dirigida a una composición de éter de celulosa en la forma de una cápsula. Ninguna de las anterioridades citadas por ese Despacho enseña una composición en la forma de una cápsula y por lo tanto se puede concluir que la misma es novedosa frente a los documentos D1 a D3.

Adicionalmente, ninguno de los documentos citados divulga ni sugiere someramente que el contenido de turbiedad en cápsulas de éter de celulosa pueda reducirse de forma significativa al limitar el contenido de cloruro de sodio remanente en el producto de éter de celulosa a partir del cual se forman las cápsulas. Así las cosas, el objeto de la presente invención es completamente novedoso frente al arte previo citado por ese Despacho pues ninguna de las anterioridades contiene todos y cada uno de los elementos reivindicados en el nuevo

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pliego que aquí presento, por lo que considero que el mismo cumple con el requisito de novedad de acuerdo con la Decisión 486.

3.2. Nivel inventivo. Respecto a la objeción hecha a este requisito de patentabilidad, quiero exponer a continuación los aspectos que demuestran la existencia de nivel inventivo de la presente solicitud.

Como ya se explicó anteriormente, ninguno de los documentos citados divulga ni sugiere someramente que el contenido de turbiedad en cápsulas de éter de celulosa pueda reducirse de forma significativa al limitar el contenido de cloruro de sodio remanente en el producto de éter de celulosa a partir del cual se forman las cápsulas. Ahora bien, dichas anterioridades tampoco sugieren la manera en que una persona versada en la materia hubiera podido obtener de manera obvia la composición reivindicada pues no había guía alguna para saber que la formación de turbiedad en las cápsulas de éter de celulosa se reduciría significativamente al limitar la cantidad de cloruro de sodio que queda en el producto de éter de celulosa a partir del cual se producen las mencionadas cápsulas.

Por otra parte, ninguno de los documentos D4 y D5 se refieren específicamente al fenómeno de la formación de turbiedad. Ninguno de los documentos citados sugiere en un proceso limitar el contenido de cloruro de sodio a 0,3% o menos con el fin de evitar la turbiedad en cápsulas de éter de celulosa. Es decir, ninguna de las enseñanzas divulgadas en dichos documentos, ni solas ni en combinación, hubieran guiado de forma directa y evidente a una persona versada en la materia al proceso aquí reivindicado.

Por lo tanto, considero que el pliego reivindicatorio que aquí presento cumple con el requisito de nivel inventivo de acuerdo con la Decisión 486.

Por las razones anteriormente expuestas, le solicito se sirva concederle el privilegio de patente a la presente solicitud pues la misma cumple con todos los requisitos necesarios de

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patentabilidad.

4. Puesto que se han atendido a cabalidad las observaciones hechas por su Despacho sobre la presente solicitud de patente, atentamente solicito se continúe con su tramitación.

Bogotá, 29 AGO. 2005

Señora Jefe,



EMILIO FERRERO

T.P.A. No. 31.929

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COLO 13518-803-001-3
MRE/MRE/
ID-001869/WPCL0286

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