

151

SUPERINDUSTRIA Y COMERCIO
Radicación : 04056932 00000002 Folios: 48
Fecha (HNB): 2004-08-25 11:57:32
Trámite : 011 PCT-PATENT 379 FASE NRCI 444 REPUESTA
Dependencia: 2820 DIVISION DE NUEVAS CREACIONES

Señores
SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO
DIVISIÓN DE SIGNOS DISTINTIVOS

E. S. D.

Ref.: Expediente No. 04-056.932
Fase Nacional Solicitud PCT No. PCT/EP02/14371.

Título: ADICION DE INICIADORES ORGÁNICOS DURANTE LA CAIDA DE PRESION
EN LA POLIMERIZACION DE MONOMEROS DE CLORURO DE VINILO.

Solicitante: Akzo Nobel N.V.

Darío Cárdenas Navas, identificado como aparece al pie de mi firma, atentamente me dirijo a ese Despacho dando respuesta al Oficio No. 6437 de fecha 9 de Julio de 2004 en relación con el asunto de la referencia.

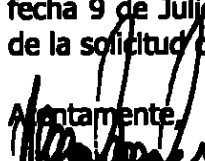
De acuerdo con lo solicitado me permito anexar copia auténtica y debidamente legalizada del documento en donde consta la cesión del derecho de la patente del inventor al solicitante junto con la Traducción Oficial correspondiente.

Igualmente, pese a no haber sido solicitadas por la Oficina de Patentes, y a manera de información adjunto a la presente copia de los documentos de prioridad de las siguientes patentes:

- Patente Europea No. 02077.471.7 de fecha 18 de Junio de 2002.
- Patente de los Estados Unidos No. 60/342.434 de fecha 21 de Diciembre de 2001.

Habiendo dado cumplimiento a los requerimientos incluidos en el Oficio No. 06437 de fecha 9 de Julio de 2004 atentamente solicito a ese Despacho proceda con la publicación de la solicitud de patente en referencia.

Atentamente,


Darío Cárdenas Navas.
c.c. 17.066.629 de Bogotá
t.p. 1.250 Minjusticia

ASSIGNMENT

Between the undersigned, MEULENBRUGGE, Lambertus a Dutch citizen domiciled at De Gemeint 11, NL-6931 HZ Westervoort, The Netherlands ; VANDUFFEL, Koen, Antoon, Kornelis a Belgian citizen domiciled at Stevinhof 18, NL-7424 AH Deventer, The Netherlands ; and WESTMIJZE, Hans a Dutch citizen domiciled at Burg. Boreellaan 1 NL-7437 BB Bathmen, The Netherlands, henceforth the Assignor, and SCHALKWIJK, Pieter Cornelis and VAN DEURSEN, Petrus Hubertus, acting in their capacity of Holders of Proxy of Akzo Nobel N.V., a company legally constituted and in existence in accordance to the laws of The Netherlands, and domiciled at Velperweg 76, NL-6824 BM Arnhem, The Netherlands, henceforth the Assignee and,

A. Whereas the Assignee has applied for registration before the Colombian Patent Office, the National Phase of the following PCT patent application:

Title of the invention	File No.	Filing date
ADDITION OF ORGANIC INITIATORS DURING THE PRESSURE DROP IN VINYL CHLORIDE MONOMER POLYMERIZATION REACTIONS		

B. Whereas the Assignors are the inventors of the invention which is the object of the above-mentioned patent application.

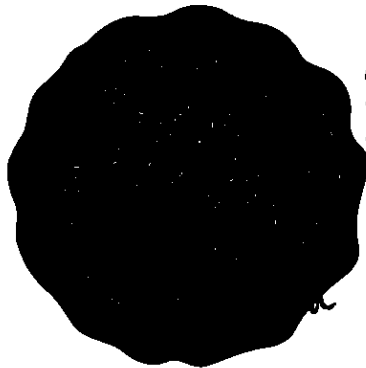
C. Whereas the Assignee is interested in obtaining the assignment of the above-mentioned patent invention, and the Assignors are interested in assigning it to the Assignee.

The following agreement has been entered:

First: The Assignors assign to the Assignee every and all rights belonging to the Assignors regarding the invention that is the object of the patent application mentioned in letter A. of this document.

Second: The Assignee accepts the aforementioned assignment





The undersigned, *Mr. M. Schellingerhout*
civil-law notary, residing at Arnhem,
the Netherlands, certifies the above
signature to be the genuine signature of
L. M. van der Grinten, H. Westmijze, H. A. M. J. van der Grinten
P. C. Schellingerhout, P. H. van der Grinten
Thus signed at Arnhem, this 27th day of
July 2004.



APOSTILLE
(Convention de la Haye de 5 octobre 1961)



1. Country : The Netherlands
2. This public document
has been signed by : M. Schellingerhout
3. acting in the capacity of : Civil law notary
4. bears the seal/stamp of : Mr. M. Schellingerhout
* Notaris te Arnhem *

CERTIFIED

5. at Arnhem, 6. at the 27 July 2004
7. by the registrar of the court
(ARRONDISSEMENTSRECHTBANK)
8. no. 04/654



9. Stamp

10. Signature: J.M.C. van den Boven

Jm van den Boven
Kalverstraat

Third: This assignment shall inure to and be binding on the parties, and their subsidiaries, affiliates, successors, assignees, and licenses, if any.

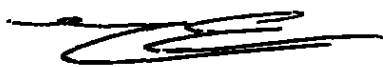
Signed on the _____ day of _____ of 2.004, by



MEULENBRUGGE, Lambertus



WESTMIJZE, Hans



VANDUFFEL, Koen, Antoon, Kornelis

Akzo Nobel N.V.

(Assignee)

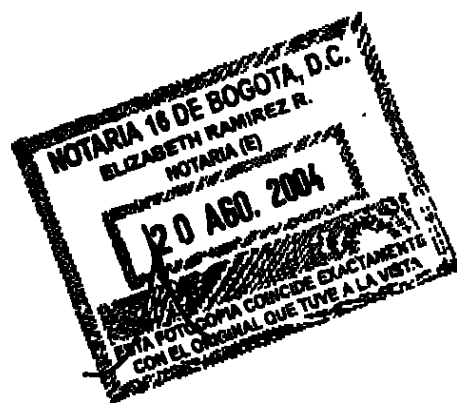


SCHALKWIJK, Pieter Cornelis
(Holder of Proxy)



VAN DEURSEN, Petrus Hubertus
(Holder of Proxy)

(This document must be duly legalized by Apostille)





File number: 09007809 Page 00001 Limited extract

English translation of an extract from the commercial register of the Chamber of Commerce and Industries for Centraal Gelderland

Legal person:

Legal form	:Naamloze Vennootschap (Public Limited Liability Company)
Name	:AKZO Nobel N.V.
Statutory seat	:Arnhem
Incorporation deed	:08-05-1911
Deed of latest amendment of articles	:02-07-2001
Authorized capital	:EUR 1.600.019.200,00
Issued capital	:EUR 572.313.720,00
Paid up capital	:EUR 572.313.720,00
There are different classes of shares	:Consult the commercial register file
Other information	:See Dutch extract

Undertaking:

Tradename(s)	:AKZO Nobel N.V.
Address	:Velperweg 76, 6824BM Arnhem
Mailing address	:Postbus 9300, 6800SB Arnhem
Telephone number	:026-3664433
Date of establishment	:08-05-1911
Description of business conducted	:See Dutch extract
Employees	:0

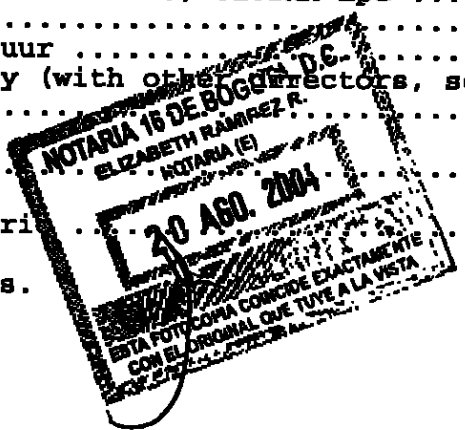
This extract contains a selection of registered incumbents

Director(s):

Name	:van der Meer, Rudolf Marinus Jacob
Date and place of birth	:27-01-1945, 's-Gravenhage
Address	:Burgemeester v d Feltzln 13, 8162MA Epe
Date of entry into office	:01-05-1993
Title	:Lid raad van bestuur
Powers	:Authorised jointly (with other directors, see articles)
Date of (present) representative authority	:01-05-1993

Name	:Strömqvist, Dag Eri
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05-07-2004	Page 00002 follows.
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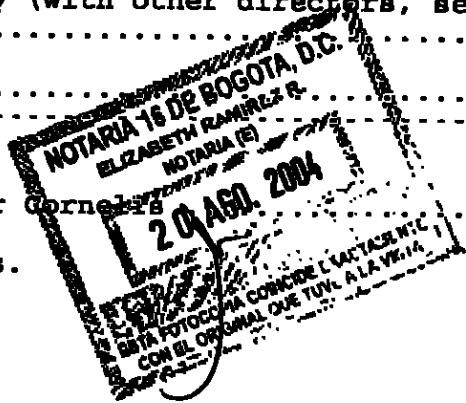
HOOFDKANTOOR
KRONENBURGSINGEL 525
POSTBUS 9292, 6800 KZ ARNHEM
T (026) 353 88 88



File number: 09007809 Page 00002 Limited extract

Date and place of birth	:22-01-1942, Skeppsholm, Sweden
Address	:Wisseloordlaan 10, 1217CS Hilversum
Date of entry into office	:01-07-2000
Title	:Lid raad van bestuur
Powers	:Authorised jointly (with other directors, see articles)
Date of (present) representative authority	:01-07-2000
Name	:Wilderbeek, Antonius Theodorus Maria
Date and place of birth	:21-01-1950, Utrecht
Address	:Coggins Lane 11, 07052 West Orange New Jersey, United States of America
Date of entry into office	:01-07-2002
Title	:Lid raad van bestuur
Powers	:Authorised jointly (with other directors, see articles)
Date of (present) representative authority	:01-07-2002
Name	:Wijers, Gerardus Johannes
Date and place of birth	:11-01-1951, Oostburg
Address	:Wilhelminapark 7, 3581NB Utrecht
Date of entry into office	:01-10-2002
Title	:Voorzitter Raad van Bestuur
Powers	:Authorised jointly (with other directors, see articles)
Date of (present) representative authority	:01-10-2002
Name	:Frohn, Robert Jozef
Date and place of birth	:12-03-1960, Nijmegen
Address	:Beekhuizeneweg 65, 6881AE Velp Gld
Date of entry into office	:01-05-2004
Title	:Lid raad van bestuur
Powers	:Authorised jointly (with other directors, see articles)
Date of (present) representative authority	:01-05-2004

Authorized signatory(signatories):	
Name	:Schalkwijk, Pieter Cornelis
05-07-2004	Page 00003 follows.



HOOFDKANTOOR
KRONENBURGSINGEL 525
POSTBUS 9292, 6800 KZ ARNHEM
T (024) 353 88 88



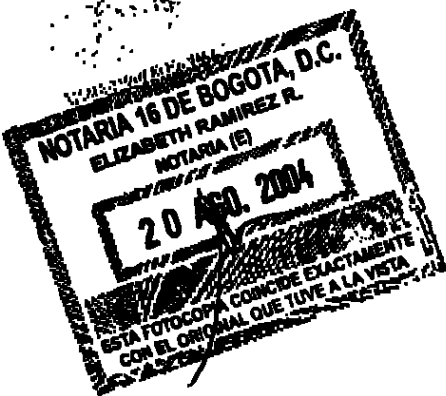
File number: 09007809	Page 00003 Limited extract
Date and place of birth	:03-08-1950, Utrecht
Address	:Zoutmanlaan 5, 6881GM Velp Gld
Function and entry into office	:Holder of power of attorney for signature, ... 01-07-1986
Title	:Octrooigevolmachtigde tevens Procuratiehouder b
Powers	:Restricted power of attorney
Commencement (present) power of attorney	:01-07-1999
Name	:van Deursen, Petrus Hubertus
Date and place of birth	:01-11-1949, Balikpapan, Indonesia
Address	:Kraijesteijnlaan 19, 6891EA Rozendaal
Date of entry into office	:01-07-1999
Title	:Octrooigevolmachtigde
Powers	:Restricted power of attorney
Commencement (present) power of attorney	:01-07-1999

Issued by the chamber of commerce

Arnhem, 05-07-2004

For extract


Mw. J.H.M. Glaap



HOOFDKANTOOR
KRONENBURGSINGEL 535
POSTBUS 9292, 6800 KZ ARNHEM
T (026) 353 88 88

The English translation of the "Description of Business conducted" is:

The establishment and exploitation of industrial companies, the trade in industrial products, the participation in other companies and all activities related thereto.





Dossiernummer: 09007809 Blad 00001 Beperkt uittreksel

Uittreksel uit het handelsregister van de Kamer van Koophandel en Fabrieken voor Centraal Gelderland

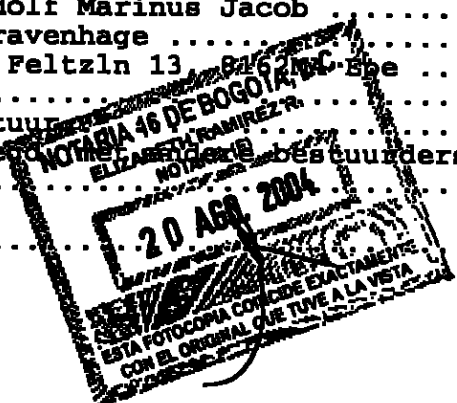
Rechtspersoon:
Rechtsvorm :Naamloze vennootschap
Naam :AKZO Nobel N.V.
Statutaire zetel :Arnhem
Akte van oprichting :08-05-1911
Akte laatste statuten-wijziging :02-07-2001
Maatschappelijk kapitaal :EUR 1.600.019.200,00
Geplaatst kapitaal :EUR 572.313.720,00
Gestort kapitaal :EUR 572.313.720,00
Er zijn verschillende soorten aandelen :Raadpleeg het handelsregisterdossier

Onderneming:
Handelsna(a)m(en) :AKZO Nobel N.V.
Adres :Velperweg 76, 6824BM Arnhem
Correspondentieadres :Postbus 9300, 6800SB Arnhem
Telefoonnummer :026-3664433
Datum vestiging :08-05-1911
Bedrijfsomschrijving :De oprichting en exploitatie van industriële ..
bedrijven, de handel in industriële produkten,
het deelnemen in andere vennootschappen en alle
werkzaamheden die met een en ander in verband ..
staan.
Werkzame personen :0

Dit uittreksel bevat een selectie van ingeschreven functionarissen

Bestuurder(s) :
Naam :van der Meer, Rudolf Marinus Jacob
Geboortedatum en -plaats :27-01-1945, 's-Gravenhage
Adres :Burgemeester v d Feltzln 13, 8162 BC Spe
Infunctietreding :01-05-1993
Titel :Lid raad van bestuur
Bevoegdheid :Gezamenlijk bevoegd bestuurs, ..
zie statuten)
Aanvang (huidige) vertegenwoordigingsbevoegdheid :01-05-1993

05-07-2004 Blad 00002 volgt.



HOOFDKANTOOR
KRONENBURGSINGEL 525
POSTBUS 9292, 6800 KZ ARNHEM
T (026) 353 88 88



Dossiernummer: 09007809 Blad 00002 Beperkt uittreksel

Naam	:Strömqvist, Dag Eric
Geboortedatum en -plaats	:22-01-1942, Skeppsholm, Zweden
Adres	:Wisseloordlaan 10, 1217CS Hilversum
Infunctietreding	:01-07-2000
Titel	:Lid raad van bestuur
Bevoegdheid	:Gezamenlijk bevoegd (met andere bestuurders, .. zie statuten)
Aanvang (huidige) vertegenwoordigingsbevoegdheid	:01-07-2000
Naam	:Wilderbeek, Antonius Theodorus Maria
Geboortedatum en -plaats	:21-01-1950, Utrecht
Adres	:Coggins Lane 11, 07052 West Orange New Jersey, Ver. Staten van Amerika
Infunctietreding	:01-07-2002
Titel	:Lid raad van bestuur
Bevoegdheid	:Gezamenlijk bevoegd (met andere bestuurders, .. zie statuten)
Aanvang (huidige) vertegenwoordigingsbevoegdheid	:01-07-2002
Naam	:Wijers, Gerardus Johannes
Geboortedatum en -plaats	:11-01-1951, Oostburg
Adres	:Wilhelminapark 7, 3581NB Utrecht
Infunctietreding	:01-10-2002
Titel	:Voorzitter Raad van Bestuur
Bevoegdheid	:Gezamenlijk bevoegd (met andere bestuurders, .. zie statuten)
Aanvang (huidige) vertegenwoordigingsbevoegdheid	:01-10-2002
Naam	:Frohn, Robert Jozef
Geboortedatum en -plaats	:12-03-1960, Nijmegen
Adres	:Beekhuizeneweg 65, 6881AE Velp Gld
Infunctietreding	:01-05-2004
Titel	:Lid raad van bestuur
Bevoegdheid	:Gezamenlijk bevoegd (met andere bestuurders, .. zie statuten)
Aanvang (huidige) vertegenwoordigingsbevoegdheid	:01-05-2004

Gevolmachtigde(n) :

05-07-2004 Blad 00003 volgt.



HOOFDKANTOOR
KRONENBURGSINGEL 525
POSTBUS 9292, 6800 KZ ARNHEM
T (026) 353 88 88



Dossiernummer: 09007809 Blad 00003 Beperkt uittreksel

Naam	:Schalkwijk, Pieter Cornelis
Geboortedatum en -plaats	:03-08-1950, Utrecht
Adres	:Zoutmanlaan 5, 6881GM Velp Gld
Functie en infunctietreding	:Procuratiehouder, 01-07-1986
Titel	:Octrooigevolmachtigde tevens Procuratiehouder b
Bevoegdheid	:Beperkte volmacht
Aanvang (huidige) volmacht	:01-07-1999
Naam	:van Deursen, Petrus Hubertus
Geboortedatum en -plaats	:01-11-1949, Balikpapan, Indonesië
Adres	:Kraijesteijnlaan 19, 6891EA Rozendaal
Infunctietreding	:01-07-1999
Titel	:Octrooigevolmachtigde
Bevoegdheid	:Beperkte volmacht
Aanvang (huidige) volmacht	:01-07-1999

Alleen geldig indien door de kamer voorzien van een ondertekening.

Arnhem, 05-07-2004

Voor uittreksel

J. H. M. Glas
Mw., J.H.M. Glas



HOOFDKANTOOR
KRONENBURGSINGEL 525
POSTBUS 9292, 6800 KZ ARNHEM
T (026) 353 88 88



The undersigned, *Mr M. Schellingerhout*
civil-law notary, residing at Arnhem,
the Netherlands, certifies the above
signature to be the genuine signature of
Mrs H.H.M. Glaap
Thus signed at Arnhem, this 2th day of
July 2004.



APOSTILLE
(Convention de la Haye de 5 octobre 1961)

1. Country : The Netherlands
2. This public document
has been signed by : M. Schellingerhout
3. acting in the capacity of : Civil law notary
4. bears the seal/stamp of : Mr. M. Schellingerhout
* Notaris te Arnhem *

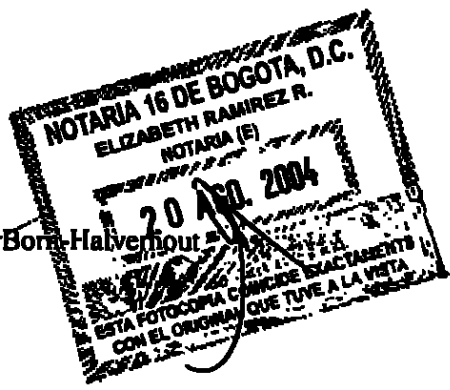
CERTIFIED

5. at Arnhem, 6. at the 27 July 2004
7. by the registrar of the court
(ARRONDISSEMENTSRECHTBANK)
8. no. 04/654



10. Signature: J.M.C. van den Born-Halverhouw

J.M.C. van den Born-Halverhouw



Traducción oficial no. 369/04 del holandés al español por Sylvia E van Uden, traductora oficial

CAMARA DE COMERCIO DE CENTRAAL GELDERLAND

Número del archivo: 09007809 Página 00001 Certificado limitado

Certificado de la Cámara de Comercio y Fábricas de Central Gelderland

Persona jurídica:

Forma jurídica	: Sociedad anónima
Nombre	: AKZO Nobel N.V.
Domicilio estatutario	: Arnhem
Escritura de Constitución	: 08-05-1911
Acta ultima modificación estatutaria	: 02-07-2001
Capital social	: EUR 1.800.019.200,00
Capital suscrito	: EUR 572.313.720,00
Capital desembolsado	: EUR 572.313.720,00
Hay diferentes clases de acciones	: Consulte el expediente del certificado mercantil

Empresa:

Razón(es) Social(es)	: AKZO Nobel N.V.
Dirección	: Velperweg 76, 6824BM Arnhem
Dirección de correspondencia	: Apartado Aereo 9300, 6800 SB Arnhem
Teléfono	: 026-3884433
Fecha de constitución	: 08-05-1911
Descripción de la empresa	: La creación y explotación de empresas industriales, el comercio de productos industriales, la participación en otras sociedades y todas las labores relacionadas al respecto.
Personas laborando	: 0

Este certificado incluye una selección de funcionarios inscritos.

Directivo(s):

Nombre	: van der Meer, Rudolf Marinus Jacob
Fecha y lugar de nacimiento	: 27-01-1945, La Haya
Dirección	: Burgemeester vd Feltz 13, 8432MA Epe
Inicio cargo	: 01-05-1993
Título	: Miembro de la Junta Directiva



Competencia : Competencia conjunta (con otros directivos, ver estatutos)

Inicio poder de representación (actual) : 01-05-1993

05-07-2004 **Página 00002 sigue**

Número del archivo: 09007809 Página 00002 Certificado limitado

Nombre	:Stromqvist, Dag Eric
Fecha y lugar de nacimiento	: 22-01-1942, Skeppsholm, Suecia
Dirección	: Wisseloordlaan 10, 1217CS Hilversum
Inicio cargo	: 01-07-2000
Título	: Miembro junta directiva
Competencia	: Competencia conjunta (con otros directivos, ver estatutos)
Inicio poder de representación (actual)	: 01-07-2000

Nombre	: Wilderbeek, Antonius Theodorus Maria
Fecha y lugar de nacimiento	: 21-01-1950, Utrecht
Dirección	: Coggins Lane 11, 07052 West Orange New Jersey, Estados Unidos
Inicio cargo	: 01-07-2002
Título	: Miembro de la Junta Directiva
Competencia	: Competencia conjunta (con otros directivos, ver estatutos)
Inicio poder de representación (actual)	: 01-07-2002

Nombre	: Wijers, Gerardus Johannes
Fecha y lugar de nacimiento	: 11-01-1951, Oostburg
Dirección	: Wilhelminapark 7, 3581NB Utrecht
Inicio cargo	: 01-10-2002
Título	: Presidente de la Junta Directiva
Competencia	: Competencia conjunta (con otros directivos, ver estatutos)
Inicio poder de representación (actual)	: 01-10-2002

Nombre	: Frohn, Robert Jozsef
Fecha y lugar de nacimiento	: 12-03-1960, Nijmegen
Dirección	: Beekhuizenseweg 65, 6881AE Gld
Inicio cargo	: 01-05-2004
Título	: Miembro de la Junta Directiva
Competencia	: Competencia conjunta (con otros directivos, ver estatutos)

Inicio poder de representación (actual) : 01-05-2004

Apoderado(s):

05-07-2004

Sigue pagina 00003

Número del archivo: 09007809 Página 00003 Certificado limitado

Nombre	: Schalkwijk, Pieter Cornelis
Fecha y lugar de nacimiento	: 03-08-1950, Utrecht
Dirección	: Zoutmanlaan 5, 6881GM Velp Gld
Cargo e inicio cargo	: Apoderado, 01-07-1988
Título	: Agente de patentes y a la vez apoderado
Competencia	: Poder limitado
Inicio poder (actual)	: 01-07-1999

Nombre	: van Deursen, Petrus Hubertus
Fecha y lugar de nacimiento	: 01-11-1949 Balikpapan, Indonesia
Dirección	: Kraljestijnlaan 19, 6891EA Rozendaal
Inicio cargo	: 01-07-1999
Título	: Agente de patentes
Competencia	: Poder limitado
Inicio poder (actual)	: 01-07-1999

Solamente válido si tiene la firma de la cámara,

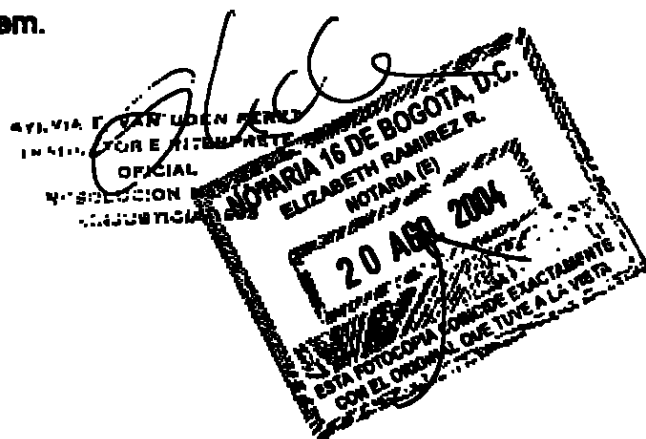
Amhem, 05-07-2004

Se firma en constancia,
[fue firmado]
Sra JHM Glaap

Al respaldo aparecen dos sellos en idioma Inglés.

Sello redondo: Juzgado del distrito de Amhem.

ES TRADUCCION FIEL Y COMPLETA.
Bogota, 15 de agosto 2004



Traducción Oficial No. 2004/484 de un documento escrito en inglés el cual para su identificación se sella con el sello del traductor al tiempo con la presente. Esta traducción ha sido preparada por Hilda Aguilera de Piedrahita, traductora oficial debidamente reconocida por el Ministerio de Relaciones Exteriores y autorizada por el Ministerio de Justicia y del Derecho por Resolución No. 323 de 1980.

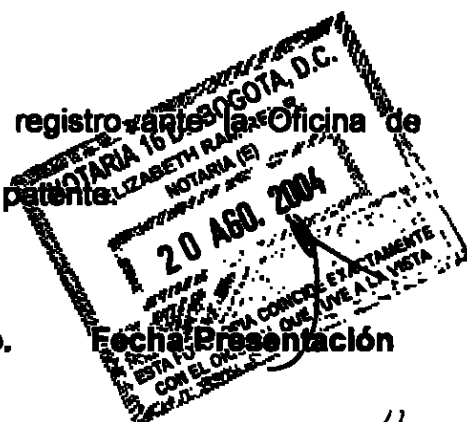
CESION

Entre los suscritos, MEULENBRUGGE, Lambertus, un ciudadano holandés con domicilio en De Gemeint 11, NL-6931 HZ Westervoort, Holanda; VANDUFFEL, Koen, Antón, Kornelis, un ciudadano belga con domicilio en Stevinhof 18, NL-7424 AH Deventer, Holanda; y WESTMIJZE, Hans, un ciudadano holandés con domicilio en Buró, Boreellaan 1 NL-7437 BB Bathmen, Holanda, en adelante los CEDENTES, y SCHALKWIJK, Pieter Cornelis y VAN DEURSEN, Petrus Hubertus actuando en sus calidades de tenedores de poder de Akzo Nobel N.V., una compañía legalmente constituida y existente de acuerdo con las leyes de Holanda, con domicilio en Velperweg 76, 6824 BM Amhem, Holanda, en adelante el CESIONARIO y,.

A. Por cuanto los CEDENTES han solicitado el registro ante la Oficina de Patentes Colombiana de la siguiente Solicitud de patente:

TITULO

Solicitud No.



Traducción Oficial No. 2004/484
HILDA AGUILERA DE PIEDRAHITA
INTERPRETE OFICIAL
MINISTERIO DE JUSTICIA

**"Adición de Iniciadores Orgánicos
durante un proceso de goteo a presión en reacciones
de polimerización de cloruro de vinilo monómero."**

**B. Por cuanto el CESIONARIO es el inventor de la invención la cual es objeto de
la anteriormente mencionada solicitud de patente.**

**C.. Por cuanto el CESIONARIO está interesada en obtener la cesión de la
Solicitud de patente anteriormente mencionada, y el CEDENTE está interesado en
cederla al CESIONARIO.**

El siguiente contrato se ha celebrado:

**PRIMERO: El CEDENTE cede al CESIONARIO todos y cada uno de los derechos
de propiedad del CEDENTE con relación a la invención la cual es objeto de la
solicitud de patente mencionada en el literal A de este documento.**

SEGUNDO: El CESIONARIO acepta la mencionada cesión.

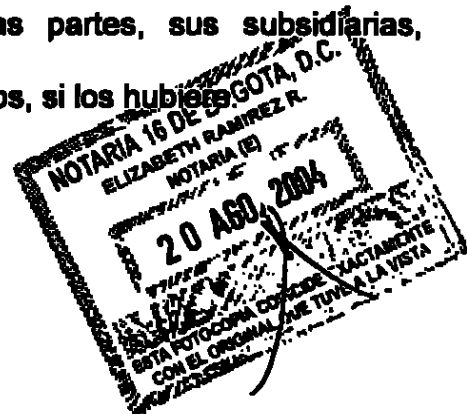
**TERCERO: Esta cesión será obligatoria para las partes, sus subsidiarias,
afiliadas, causahabientes y cesionarios y licenciatarios, si los hubiere.**

Firmado este día de de 2004.

Por. Firmado: Ilegible

MEULENBRUGGE, Lambertus

Por. Firmado: Ilegible



**TRADUCCION OFICIAL No. 3004/482
HILDA AGUILERA DE PIEDRAHITA
INTERPRETE OFICIAL
RES. No. 323 DEL MIN. DE JUSTICIA**

VANDUFFEL, Koen, Antón, Komelis

Por: Firmado: Ilegible

WESTMIJZE, Hans

Akzo Nobel N.V.

(Cesionario)

Firmado: Ilegible

Firmado: Ilegible

Pieter Cornelis Schalkwijk

Petrus Hubertus van Deursen

(Tenedor de Poder)

(Tenedor de Poder)

(Este documento deberá ser debidamente legalizado mediante Apostilla)

El suscrito, Sr. M. Schellingerhout, notario de la ley civil, residente en Arnhem, Holanda, certifica la anterior firma como firma genuina de MEULENBRUGGE, Lambertus, VANDUFFEL, Koen, Antón, Komelis, WESTMIJZE, Hans, Pieter Cornelis Schalkwijk y Petrus Hubertus van Deursen.

Por consiguiente se firma en Arnhem, este día 12 de Julio de 2004.

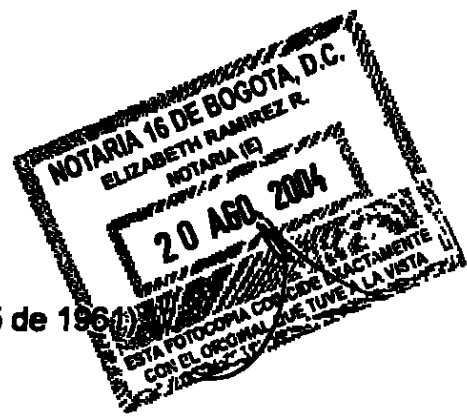
(Firmado)

(Sello de Oblea)

APOSTILLA

(Convención de la Haya de Octubre 5 de 1961)

- 1. País: Holanda



REDUCCION OFICIAL No. 2004/484
HILDA AGUILERA DE PIEDRAHITA
INTERPRETE OFICIAL
L.º No. 323 DEL MIN. DE JUSTICIA

Este documento público

- 2. Ha sido firmado por . M. Schellingerhout
- 3. Actuando en calidad de Notario Público de la Ley Civil
- 4. Lleva el sello/estampilla de . M. Schellingerhout * Notaris te Arnhem*

CERTIFICADO

- 5. En Arnhem
- 6. El 27 de Julio de2004
- 7. Por El Registrador de la Corte (arrondissementsrechtbank)
- 8. Número 04/654
- 9. Sello (Sello)
- 10. Firma (Firmado)

J.M.C. van den Born-Halverhout

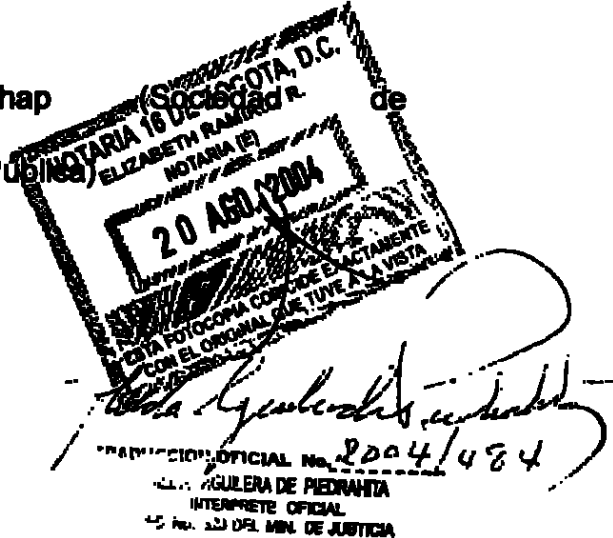
KAMER VAN KOOPHANDEL CENTRAAL GELDERLAND

Expediente Número: 0907809 Página 00001Extracto Limitado

Traducción al Inglés de un extracto del registro comercial de la Cámara de Comercio e Industria para Centraal Gelderland.

Persona Jurídica:

Forma Legal :Naamloze Vennootschap
Responsabilidad Limitada P
Nombre :Akzo Nobel N.V.
Domicilio Legal :Arnhem
Escritura de Constitución :08-05-1911



Escritura de la última

Reforma de artículos :02-07-2001
Capital autorizado :EUR 1.600.019.200,00
Capital expedido :EUR 572.313.720,00
Capital Pagado :EUR 572.313.720,00

Existen diferentes clases

De acciones :Consulte el archivo del registro comercial
Otra Información : Ver Extracto Holandés

Compromiso:

Nombre(s) Comercial(es) : Akzo Nobel N.V.
Dirección :Velperweg 76, 6824BM Arnhem
Dirección Postal :Postbus 9300, 6800SB Arnhem
Número Telefónico :026-3664433
Fecha de establecimiento :08-05-1911

Descripción del negocio: Ver Extracto en holandés

Empleados :0

Este extracto contine una selección de cargos registrados

Director(es):

Nombre :van der Meer, Rudolf Marinus Jacob

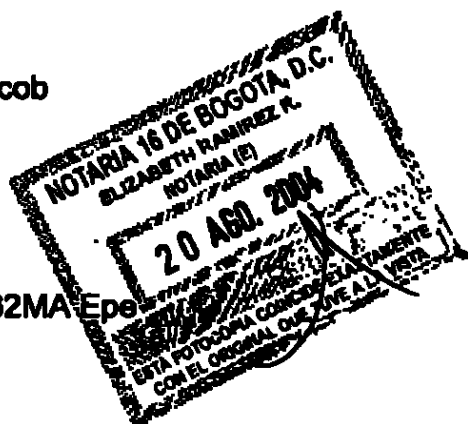
Fecha y Lugar de

Nacimiento :27-01-1945, 's-Gravenhage

Dirección :Burgemeester v d Feltzn 13, 8162MA Epe

Fecha Entrada a

La Oficina :01-05-1993



TRADUCCION OFICIAL No. 2004/484
HILDA AGUILERA DE PIEDRANTIA
INTERPRETE OFICIAL
RES. No. 323 DEL MIN. DE JUSTICIA

Cargo :Lid raad van bestuur
Facultades :Autorizado conjuntamente (ver otros directores, ver articulo)
Fecha de la facultad
Representativa
(presente) :01-05-1993
Nombre :Stromqvist, Dag Eric
05-07-2004 Sigue Página 00002.

KAMER VAN KOOPHANDEL CENTRAAL GELDERLAND

Expediente Número: 09007809 Página 00002Extracto Limitado

Fecha y Lugar

De Nacimiento: 22-01-1942, Skeppsholm, Suecia
Dirección :Wisseloordlaan 10, 1217CS Hilversum
Fecha Entrada a

La Oficina :01-07-2000
Cargo : Lid raad van bestuur
Facultades :Autorizado conjuntamente (ver otros directores, ver articulo)

Fecha de la facultad

Representativa

(presente) :01-07-2000
Nombre :Wilderbeek, Antonius Theodorus Maria

Fecha y Lugar

De Nacimiento :21-01-1950, Utrecht
Dirección :Coggins Lane 11, 07052 West Orange New Jersey, Estados Unidos de América



COADUCCION OFICIAL No. 2004/422
HILDA AGUILERA DE PIEDRAHITA
INTERPRETE OFICIAL
LEY No. 123 DEL MIN. DE JUSTICIA

Entrada

A la Oficina :01-07-2002

Cargo: Lid raad van bestuur

Facultades : Autorizado conjuntamente (ver otros directores, ver articulo)

Fecha de la facultad

Representativa

(presente) :01-07-2002

Nombre :Wijers, Gerardus Johannes

Fecha y Lugar

De Nacimiento :11-01-1951, Oostburg

Dirección :Wilhelminapark 7, 3581NB Utrecht

Fecha Entrada

A la Oficina :01-10-2002

Cargo: Voorzitter Raad van Bestuur

Facultades : Autorizado conjuntamente (ver otros directores, ver articulo)

Fecha de la facultad

Representativa

(presente) :01-10-2002

Nombre :Frohn, Robert Jozef

Fecha y Lugar

De Nacimiento :12-03-1960, Nijmegen

Dirección :Beekhuizenseweg 65, 6881AE Velp Gl

Fecha Entrada

A la Oficina :01-05-2004



TRADUCCION OFICIAL No. 2004/1280
HILDA AGUILERA DE PIEDRAHITA
INTERPRETE OFICIAL
RES. No. 323 DEL MIN. DE JUSTICIA

123

Cargo: Lid Raad van Bestuur
Facultades : Autorizado conjuntamente (ver otros directores, ver articulo)

Fecha de la facultad

Representativa

(presente) :01-05-2004

Signatario Autorizados (signatarios):

Nombre :Schalkwijk, Pieter Cornelis

05-07-2004 Sigue Página 00003

KAMER VAN KOOPHANDEL CENTRAAL GELDERLAND

Expediente Número: 09007809 Página 00003 Extracto Limitado

Fecha y Lugar

De Nacimiento :03-08-1950, Utrecht

Dirección :Zoutmanlaan 5, 6881GM Velp Gld

Función y Entrada

A la Oficina :Tenedor de Poder para firma, 01-07-1986

Cargo: Octrooigevolmachtigde tevens Procuratiehouder b

Facultades :Restringidas al Poder

Comienzo (presente)

Del Poder :01-07-1999

Nombre :vanDeursen, Petrus Hubertus

Fecha y Lugar

De Nacimiento :01-11-1949, Balikpapan, Indonesia

Dirección :Kraijesteijnlaan 19, 6891EA Rozendaal

Fecha Entrada



TRADUCCION OFICIAL No. 2004/484
HILDA AGUIRRE DE PIEDRAHITA
INTERPRETE OFICIAL
C.E. No. 323 DEL MIN. DE JUSTICIA

23

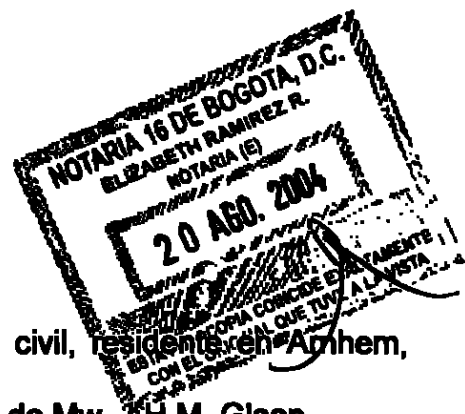
A la Oficina :01-07-999
Cargo: Octrooigevolmachtigde
Facultades :Restringidas al Poder
Comienzo (presente)
Del Poder :01-07-1999
Expedido por la Cámara de Comercio
Sello
Amhem, 05-07-2004
Para Extracto
(Firma)
Mw. J.H.M. Glaap

La traducción al inglés de "Descripción del Negocio Conducido" es:

El establecimiento y explotación de compañías industriales, el comercio de productos industriales, la participación en otras compañías y todas las actividades relacionadas con ellas.

En Holandés el extracto del registro comercial.

El suscrito, Sr. M. Schellingerhout, notario de la ley civil, residente en Amhem, Holanda, certifica la anterior firma como firma genuina de Mw. J.H.M. Glaap



TRADUCCION OFICIAL No. 2004 682
HILDA AGUILERA DE PEDRAHITA
LEY NOT. CIVIL
No. 1 de 1992, Art. 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43, 44, 45, 46, 47, 48, 49, 50, 51, 52, 53, 54, 55, 56, 57, 58, 59, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 71, 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83, 84, 85, 86, 87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, 98, 99, 100

Por consiguiente se firma en Arnhem, este día 27 de Julio d 2004.

(Firmado)

(Sello de Oblea)

APOSTILLA

(Convención de la Haya de Octubre 5 de 1961)

1. País: Holanda

Este documento público

2. Ha sido firmado por . M. Schellingerhout

3. Actuando en calidad de Notario Público de la Ley Civil

4. Lleva el sello/estampilla de . M. Schellingerhout * Notaris te Arnhem*

CERTIFICADO

5. En Arnhem

6. El 27 de Julio de 2004

7. Por El Registrador de la Corte (arrondissementsrechtbank)

8. Número 04/854

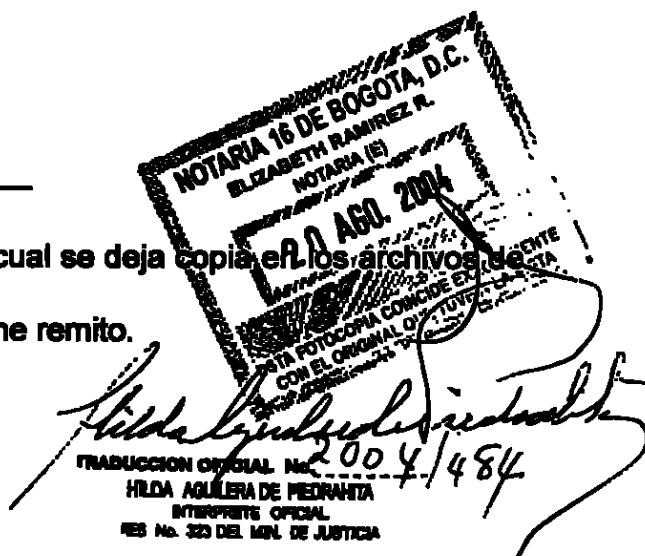
9. Sello (Sello)

10. Firma (Firmado)

J.M.C. van den Born-Halverhout

ES TRADUCCIÓN FIEL Y COMPLETA de la cual se deja copia en los archivos de esta oficina para su confrontación y a la cual me remito.

Bogotá, D.C., Agosto 12 de 2004.





MINISTERIO DE RELACIONES
EXTERIORES

firmado
655778

CUYA FIRMA APARECE EN EL
DOCUMENTO DESEMPEÑA
LAS FUNCIONES INDICADAS

NO SE Asume
RESPONSABILIDAD DEL TEXTO

2004 AUG 18 P 1:27

JEFE DE LEGALIZACIONES
SANTAFE DE BOGOTA D.C.



**Europäisches
Patentamt**

**European
Patent Office**

**Office européen
des brevets**

Bescheinigung

Certificate

Attestation

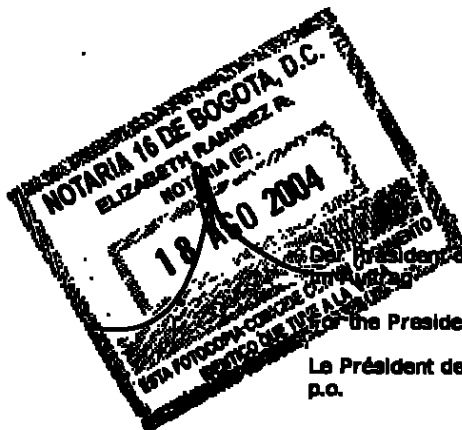
Die angehefteten Unterla-
gen stimmen mit der
ursprünglich eingereichten
Fassung der auf dem näch-
sten Blatt bezeichneten
europäischen Patentanmel-
dung überein.

The attached documents
are exact copies of the
European patent application
described on the following
page, as originally filed.

Les documents fixés à
cette attestation sont
conformes à la version
initialement déposée de
la demande de brevet
européen spécifiée à la
page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

02077471.7



President des Europäischen Patentamts
the President of the European Patent Office
Le Président de l'Office européen des brevets
p.o.

R C van Dijk



Anmeldung Nr:
Application no.: 02077471.7
Demande no:

Anmeldetag:
Date of filing: 18.06.02
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

Akzo Nobel N.V.
Velperweg 76
6824 BM Arnhem
PAYS-BAS

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se référer à la description.)

Addition of organic initiators during a pressure drop in vinyl chloride monomer polymerization reactions

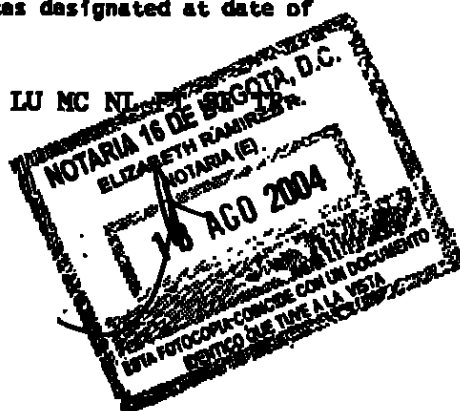
In Anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s)
revendiquée(s)
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/
Classification internationale des brevets:

C08F14/00

Am Anmeldetag benannte Vertragsstaaten/Contracting states designated at date of
filing/Etats contractants désignées lors du dépôt:

AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU MC NL



178

ACD 2943 PDEP

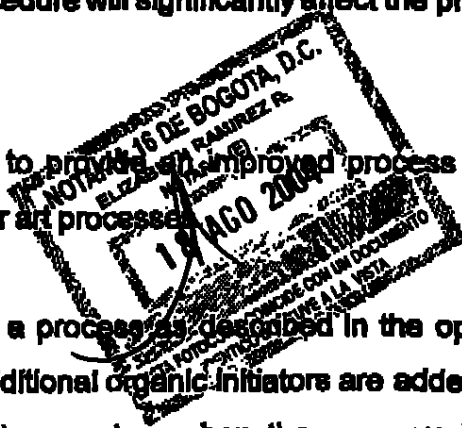
ADDITION OF ORGANIC INITIATORS DURING A PRESSURE DROP IN VINYL CHLORIDE MONOMER POLYMERIZATION REACTIONS

- 6 The present invention relates to a process wherein at least one organic initiator is used to polymerize a mixture containing one or more monomers of which one is vinyl chloride monomer (VCM).

Such a process can be performed in many different ways as is known to a person skilled in the art. It is generally desired to shorten the total polymerization time. This can be achieved by shortening the period of constant pressure in the polymerization reactor, or this can be achieved by reducing the time required to complete the polymerization reaction after the pressure in the reactor has dropped. An example of a process wherein the time of pressure drop is reduced, is US 5,908,905, wherein a process is described, which is completed at a higher temperature than a preset temperature for the polymerization. However, such a temperature increase may result in discoloration of the polymer. Above all, it has the disadvantage of influencing the molecular weight of the polymers formed. US 5,739,222 describes an other process, wherein, when the internal pressure of the reactor declines, the remainder of the overall monomer charge is continuously fed to the reactor. This procedure will significantly affect the physical properties of the polymer particles.

It is an object of the present invention to provide an improved process which overcomes the disadvantages of the prior art processes.

The process of the present invention is a process as described in the opening paragraph, and wherein one or more additional organic initiators are added to a polymerization mixture at the reaction temperature when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride



28

ACD 2943 PDEP

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monomer, with the proviso that the polymer formed is not subject to radio frequency dielectric heating in the presence of additional organic initiator.

During VCM polymerizations, the pressure initially is proportional to the temperature of the reaction mixture. This is because the pressure in the polymerization reactor is predominantly determined by the vapour pressure of the VCM in the polymerization mixture. Towards the end of the polymerization reaction, the VCM is depleted. At a certain moment no liquid VCM phase is present any longer and the pressure in the reactor starts to drop below the otherwise observed pressure at that temperature. After and/or during the pressure drop there is still VCM present in the gas phase, but there is typically more and more being adsorbed in the water and polymer phase of the reaction mixture. At that time the polymerization rate conventionally reduces and the cooling capacity of the reactor is not optimally used.

15

The inventors have now identified these problems and found that the available cooling capacity of the reactor can be used more efficiently if the polymerization rate is enhanced by addition of one or more additional organic initiators after and/or during the pressure drop. Preferably, the additional initiator is added on or after the moment at which the pressure drops in the reactor. It is less preferred to add said additional initiator before that moment as it may lead to an undesirable runaway reaction which is generally accompanied by an undesirable increase in pressure. Apart from the more efficient use of the cooling capacity, the total polymerization time is reduced due to a higher pressure drop rate, thereby shortening the period in which the pressure of the reactor is dropping. Also, the reactor space-time yield could be improved.

It is noted that US 4,015,065 describes a process wherein polymerization initiator is added at the pressure drop to the polymer formed, while subjecting the

179

ACD 2943 PDEP

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polymer to radio frequency dielectric heating. With this process, it is aimed to remove or partially remove the abundantly present vinyl halide monomer from the vinyl halide polymer. As mentioned above, the present invention solves the problem of a relatively large contribution of the pressure drop time to the overall polymerization time, which is completely different from the problem solved in US 4,015,065. Also, the use of radio frequency dielectric heating is less desired since it may destabilize and/or discolor the polymer that is produced.

With the term "after and/or during the pressure drop" it is meant the time during which the pressure in the polymerization reactor drops, including the 30 minutes, preferably 20 minutes, more preferably 10 minutes and most preferably 5 minutes before the pressure drop is actually observed. Preferably the addition of initiator takes place after the pressure was dropping below the pressure that was observed earlier at the same temperature.

15

The additional initiators can be any kind of organic peroxide suitable for use in the polymerization process, such as peroxydicarbonates and acetyl-cyclohexyl-sulfonyl-peroxide. Hence, the additional initiator can be the same as or different from the initiator(s) used in the polymerization before the pressure drop. More preferred additional initiators are fast initiators such as organic peroxides having a half-life of from 0.0001 to 1 hour at the polymerization temperature. Even more preferred are peroxides having a half-life ranging from 0.0001 to 0.5 hour at the polymerization temperature. Most preferred are extremely fast peroxides having a half-life of from 0.0001 to 0.05 hour at the polymerization temperature.

Preferred examples of organic peroxides used in the process of the invention are 1,1,3,3-tetramethylbutylperoxy methoxy acetate, tert-butylperoxy methoxy acetate, tert-amylperoxy methoxy acetate, tert-butylperoxy ethoxy acetate, diisobutanoylperoxide (Trigonox® 187), hexanoyl-dodecyl peroxide, 2-ethylbutanoyl-isononanoyl peroxide, isobutanoyl-lauroyl peroxide, isobutanoyl-

29

ACD 2943 PDEP

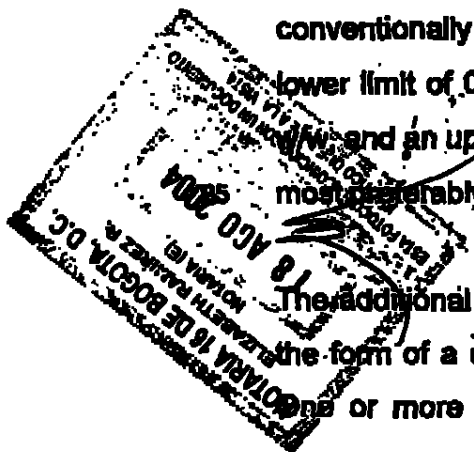
4

isononanoyl peroxide, bis(tert-butylperoxy) oxalate, cyclododecyl-
tert-butylperoxy oxalate, 2,2-bis-2-ethylhexanoylperoxy-4-methylpentane, 2,2-
bis-2-ethylbutanoylperoxy-4-methylpentane, 2,2-bis(2,2-
dimethylpropanoylperoxy)-4-methylpentane, 1-(2-ethylhexanoylperoxy)-1,3-
5 dimethylbutyl-1-peroxyphthalate (Trigonox® 267) or 2,4,4-trimethylpentyl-2-
peroxyneodecanoate (Trigonox® 423), tert-amyl peroxyneodecanoate
(Trigonox® 123), tert-butyl peroxyneodecanoate (Trigonox® 23), benzene
(m,p)di(2-isopropyl-2-peroxyneodecanoate), 2-methyl-4-hydroxypentane-2-
peroxyneodecanoate, α -cumyl peroxyneodecanoate (Trigonox® 99), and
10 peroxydicarbonates such as di-sec-butylperoxydicarbonate (Trigonox® SBP),
di(4-tert-butylcyclohexyl)peroxydicarbonate (Perkadox® 16) and di(2-ethylhexyl)
peroxydicarbonate (Trigonox® EHP).

Other organic peroxides having a half-life ranging from 0.0001 to 1 hour at
polymerization temperature can also be used. Whether a peroxide is suitable and
15 has the required half-life can be determined by conventional thermal
decomposition studies in monochlorobenzene, as is well-known in the art (see for
instance the brochure "Initiators for high polymers" with code 10013921001
obtainable from Akzo Nobel).

20 The amount of additional initiator (or initiators) to be used in a process according
to the invention after and/or during the pressure drop is within the range
conventionally used in polymerization processes. Typically, this range has a
lower limit of 0.001% w/w of additional initiator and more preferably of 0.005%
w/w and an upper limit of 0.3% w/w of additional initiator, preferably of 0.2% w/w;
most preferably, 0.05% w/w, based on the weight of VCM monomer, is used.

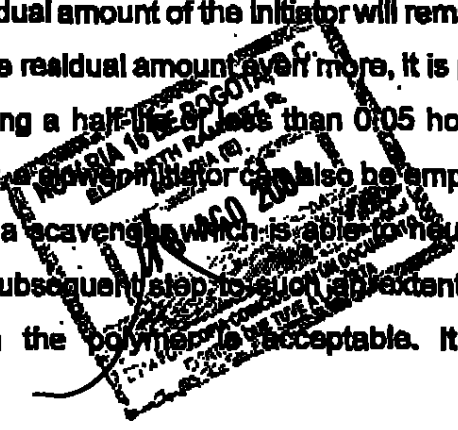
The additional initiator is added to the reactor in the pure form or, preferably, in
the form of a dilute solution or dispersion (such as a suspension or emulsion).
One or more suitable solvents can be used to dilute the additional initiator.



180

ACD 2943 PDEP

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- Preferably, such solvents are easily removed during the steps where the polymer is worked up after the polymerization process, such as alcohols, or they are of such a nature that it is acceptable to leave them as a residue in the final polymer, as in the case of solvents that are desired plasticizers for the final resin.
- 6 Furthermore, it can be advantageous, but is not necessarily required, that such solvents do not adversely affect the thermal stability of the Initiator dissolved therein, as can be verified by analyzing the half-life temperature of the Initiator in said solvent. An example of such a solvent is isododecane. If an Initiator dispersion is added, then the dispersion can be of either the initiator itself or of a
- 10 solution of the Initiator, preferably in said suitable solvents. Preferably, the dispersion is an aqueous dispersion. Preferably, the initiator is added in a concentration of at least 0.1% w/w, more preferably 0.5% w/w, and most preferably 2% w/w, and at most 75% w/w, more preferably at most 60% w/w, even more preferably at most 50% w/w, even more preferably 25% w/w, and
- 15 most preferably 15% w/w. The more dilute Initiator solutions or dispersions ensure rapid mixing of the initiator and the polymerization mixture, which leads to a more efficient use of the initiator, which is important for the fast initiators that are used.
- 20 The addition after and/or during the pressure drop preferably is the addition of fast initiators having a half-life of less than 1 hour at the polymerization temperature, as a relatively small residual amount of the Initiator will remain in the polymer formed. In order to reduce the residual amount even more, it is preferred to add extremely fast peroxides having a half-life of less than 0.05 hour at the
- 25 polymerization temperature. However, a slower initiator can also be employed. In that case it can be preferred to add a scavenger which is able to neutralize or destroy the residual initiator in any subsequent step to such an extent that the residual amount of the initiator in the polymer is acceptable. It is also
- 

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ACD 2943 PDEP

6

contemplated to add a scavenger when fast and/or extremely fast peroxides are used.

During the first part of the polymerization reaction any conventional initiating system can be used. Herein, the initiator can be added intermittently or continuously to the polymerization mixture at the polymerization temperature. All of the initiator can be added to the reactor at once in just one portion. In such case, preferably a relatively slow initiator having a half-life of more than 1 hour is used. The initiator may also be added at at least 2, preferably at least 4, more preferably at least 10, and most preferably at least 20 moments.

Typically, one or more protective colloids are used in the polymerization process of the invention. Examples of suitable protective colloids are protective colloids such as polyvinyl alcohols (PVAs), which may, for example, be (partially) saponified polyvinyl acetates with a degree of hydrolysis of at least 40%, more preferably 60%, and most preferably 62%, and a degree of hydrolysis of at most 90%, more preferably 85%, and most preferably 80%. If for example two PVAs are employed, both PVAs may have a similar degree of hydrolysis. It may also be envisaged that the two PVAs have a different degree of hydrolysis. Although said PVAs are the preferred protective colloids for processes according to the invention, it is also possible to use other conventional protective colloids, such as cellulose, water-soluble polymers, oil-soluble emulsifying agents or water-soluble emulsifying agents. It is also envisaged to employ a combination of two or more of the above protective colloids. The amount and type of colloid that is to be used is well-known and conventional.

Optionally, at least one surfactant can be added at the same time as the additional initiator to the reaction mixture so as to reduce the (excessive)



181

ACD 2943 PDEP

7

formation of foam. Examples of such surfactants are polyvinyl acetates with a degree of hydrolysis of less than 80%, and conventional foam reducers as generally known to a person skilled in the art. Preferably, the surfactant is added together with the additional initiator.

5

Preferably, the addition of the initiator and optionally of the surfactant, either during the constant pressure time or after and/or during the pressure drop, can be effected at any suitable entry point to the reactor. Such an entry point may be positioned below or above the surface of the reaction mixture, as desired. The

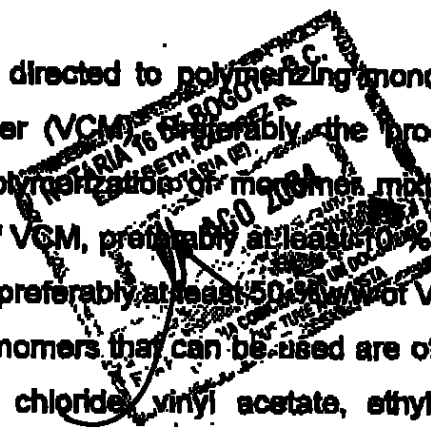
10 Initiator and the protective colloid can be added separately to the reactor via two individual entry points, or they can be pre-mixed and enter the reactor through one entry port. If water is added in the course of the polymerization process, for example to compensate for the shrinkage of the reactor content due to the polymerization reaction, it can be advantageous to use the line through which this

15 water is added to also add the initiator and/or the protective colloid. It can also be envisaged that the reactor comprises dedicated piping and a plurality of entry points located in different positions on the periphery of the said reactor, so as to ensure better, faster, and more homogeneous mixing of the initiator and/or the protective colloid in the reaction mixture. If the reactor further comprises a reflux

20 condensor, it is contemplated to add the initiator and/or the protective colloid to the reaction mixture via the reflux condensor through one or more entry points.

The process according to the invention is directed to polymerizing monomer mixtures comprising vinyl chloride monomer (VCM). Preferably, the process

25 according to the invention involves the polymerization of monomer mixtures comprising at least 5% by weight (%w/w) of VCM, preferably at least 10% w/w, more preferably at least 20 %w/w, and most preferably at least 50 %w/w of VCM, based on the weight of all monomer. Comonomers that can be used are of the conventional type and include vinylidene chloride, vinyl acetate, ethylene,



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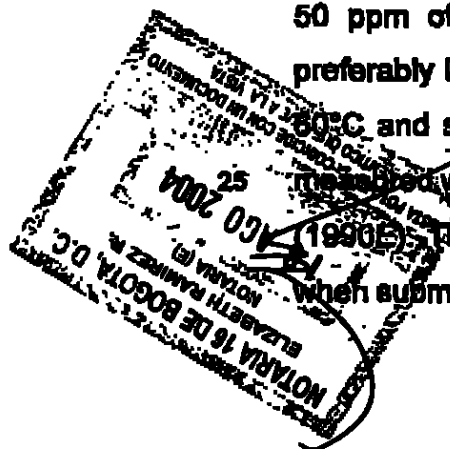
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propylene, acrylonitrile, styrene, and (meth)acrylates. More preferably, at least 80% w/w of the monomer(s) being polymerized is made up of VCM, while in the most preferred process the monomer consists essentially of VCM. As is known in the art, the polymerization temperature of such processes to a large extent
5 determines the molecular weight of the resulting resin.

The polymerization process can be conducted as a mass process wherein the reaction mixture is predominantly monomer, or as a more preferred suspension process wherein the reaction mixture typically is a suspension of monomer in
10 water, or as an emulsion or micro-emulsion process wherein the monomer typically is emulsified in water. In these processes the usual additives will have to be used. For example, if the monomer is present in the form of a suspension in water, the usual additives like surfactant(s), anti-fouling agent(s), pH-buffer(s), etc. may be present. Depending on the type of polymer desired, each of the
15 above-mentioned processes may be preferred. The process according to the invention is especially suited for use in suspension processes.

After the polymerization, the resulting (co)polymer (or resin) will be worked up as is usual in the art. Polymers obtained by a suspension polymerization according
20 to the invention, for example, will be submitted to the usual drying and screening steps. The resulting resin preferably is characterized in that it contains less than 50 ppm of residual initiator, more preferably less than 40 ppm, and most preferably less than 25 ppm of initiator. Immediately after drying for 1 hour at 60°C and screening. The resin was found to exhibit excellent heat stability as measured with a Metrastat® PSD280 testing oven in accordance with ISO 182-2 (1990). The improved heat stability proved that the resin hardly discoloured when submitted to melt-processing steps, e.g., to form shaped articles.



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ACD 2943 PDEP

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Examples to demonstrate the unexpected effect of the addition of fast peroxides during the pressure drop in PVC suspension polymerization.

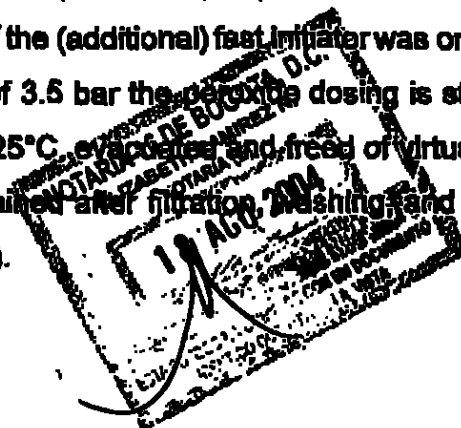
Experimental

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In a standard suspension polymerization experiment, a temperature controlled 1-liter stainless steel Büchi reactor provided with one baffle, a three-bladed stirrer, a pressure transducer, a VCM feed line, a nitrogen purge line, was charged with 425 g demineralized water, Alcotex B72 (1000 ppm on VCM) in an 5% w/w solution in demineralized water and was pressurized to 15 barg, using nitrogen. If
10 no leaks are observed, the reactor is evacuated and pressurized with nitrogen up to 5 barg three times to flush out virtually all air. Next, the reactor was evacuated and charged with 250 g VCM ex Akzo Nobel Salt & Basics, followed by heating up of the reactor to polymerization temperature in 30-60 min.

15 The conventional peroxide such as Trigonox® EHP (Tx EHP), used to initiate the polymerization, is added via a hot start procedure. The hot start procedure means that the peroxide is added to the reactor at polymerization temperature in a very short period, i.e. 1-5 min.

During the pressure drop period of the polymerization a fast initiator with a half-life at polymerization temperature of less than 0.1 hrs, is added in the reactor until
20 the pressure in the reactor has dropped 3.5 bar. For adding of the (additional) fast initiator, a relatively lowly concentrated (about 1% w/w) emulsion is used. In the comparative example the addition of the (additional) fast initiator was omitted. After reaching the said pressure drop of 3.5 bar the peroxide dosing is stopped
25 and then the reactor was cooled to 20-25°C, evacuated and freed of virtually all-remaining VCM. The polymer was obtained after filtration, washing, and drying (at 60°C for 1 hour using a fluidized bed).



32

ACD 2943 PDEP

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Example 1

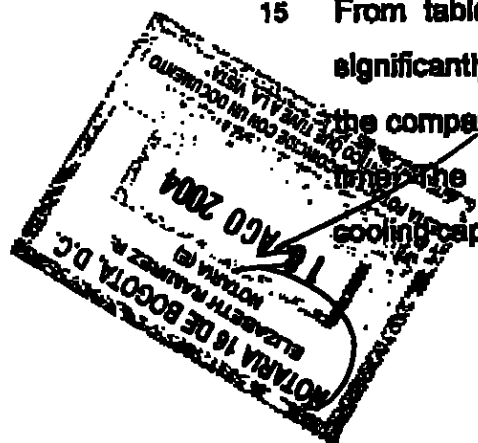
The Initiators used (Tx EHP (75% w/w) dissolved in isododecane (25% w/w), and Tx 187 [0.3% w/w aqueous emulsion]), the amount dosed, dosing procedure and the polymerization results are presented in the table 1.

After reaching the polymerization temperature the mentioned amount of Tx EHP (calculated on VCM) in isododecane is added in a period of 1-5 minutes.

Table 1; The results of VCM suspension polymerization with di(2-ethylhexyl) peroxydicarbonate (Tx EHP) at 57°C without (Comparison example) and with (Example 1) adding of diisobutanoyl peroxide (Tx 187) during the pressure drop

	Example 1	Comparison example
Hot start peroxide and quantity	800 ppm Tx EHP	800 ppm Tx EHP
Adding peroxide during pressure drop	80 ppm Tx 187 during 30 minutes	None
Constant pressure time	182 min	180 min
Pressure drop rate	7 bar/hr	4 bar/hr
Polymerization time until the reactor pressure has dropped 3.5 barg	210 minutes	230 minutes
PVC yield	91%	87%

From table 1 it is deduced that the process of Example 1 clearly has a significantly higher pressure drop rate compared to the conventional process of the comparison example. This higher drop rate leads to a shorter polymerization time. The higher pressure drop rate clearly makes for a better usage of the cooling capacity of the reactor.



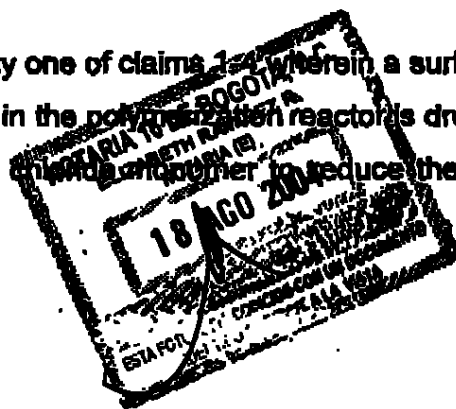
183

ACD 2943 PDEP

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Claims

1. Process wherein at least one organic initiator is used to polymerize a mixture containing one or more monomers of which one is vinyl chloride monomer, wherein at least one additional organic initiator is added to the polymerization mixture at the reaction temperature when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride monomer, with the proviso that the polymer formed is not subject to radio frequency dielectric heating in the presence of additional organic initiator.
2. A process according to claim 1 wherein the additional organic initiator, that is added after and/or during the pressure drop, has a half-life of from 0.0001 hour to 1 hour, at the polymerization temperature.
3. A process according to claim 2 wherein the additional organic initiator, that is added after and/or during the pressure drop, has a half-life of from 0.0001 hour to 0.5 hour, at the polymerization temperature.
4. A process according to any one of claims 1-3 wherein at least part of the additional organic initiator is added intermittently and/or continuously.
5. A process according to any one of claims 1-4 wherein a surfactant is also added when the pressure in the polymerization reactor is dropping due to the depletion of the vinyl chloride monomer to reduce the formation of foam.



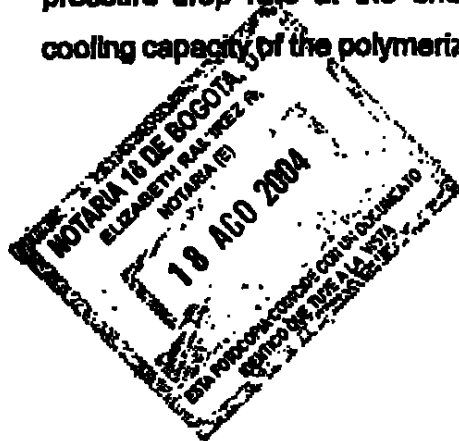
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Abstract

The invention pertains to a process to polymerize one or more monomers wherein at least one initiator and optionally at least one surfactant are added to a polymerization mixture when the pressure in the polymerization reactor is dropping. This process allows a shorter polymerization time due to a higher pressure drop rate at the end of the polymerization reaction. Moreover, the cooling capacity of the polymerization reactor is used more efficiently.



184

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APPLICATION NUMBER: 60/342,434

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M. TARVER
Certifying Officer

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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

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INVENTOR(S)					
Given Name (first and middle (if any))	Family Name or Surname	Residence (City and either State or Foreign Country)			
Hans	Westmijze	Bathmen, Netherlands			
Andreas Petrus	Van Swieten	Velp, Netherlands			
Lambertus	Meulenbrugge	Westervoort, Netherlands			
☒ Additional inventors are being named on the <u>1</u> separately numbered sheets attached hereto					
TITLE OF THE INVENTION (500 characters max)					
CONTINUOUS DOSING OF ~~EXTREMELY~~ FAST INITIATORS DURING POLYMERIZATION REACTIONS					
Direct all correspondence to: CORRESPONDENCE ADDRESS					
☐ Customer Number					
OR		Type Customer Number here		Place Customer Number Bar Code Label here	
☒ Firm or Individual Name		Ralph Mancini			
Address		AKZO NOBEL INC.			
Address		7 Livingstone Avenue			
City		Dobbs Ferry		State	New York
Country		U.S.		ZIP	10522
		Telephone	914-674-5485	Fax	914-693-4236
ENCLOSED APPLICATION PARTS (check all that apply)					
☒ Specification		Number of Pages	12	☐ CD(s), Number	
☐ Drawing(s)		Number of Sheets		☐ Other (specify)	
☐ Application Data Sheet		See 37 CFR 1.76			
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT					
☐ Applicant claims small entity status. See 37 CFR 1.27.				FILING FEE AMOUNT (\$)	
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The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.					
☒ No.					
☐ Yes, the name of the U.S. Government agency and the Government contract number are:					

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Respectfully submitted,
SIGNATURE Ralph J. Mancini
TYPED or PRINTED NAME Ralph J. Mancini
TELEPHONE 914-674-5485

Date 12/21/2001

REGISTRATION NO. 34,054
Docket Number: ACD 2916 PRUS

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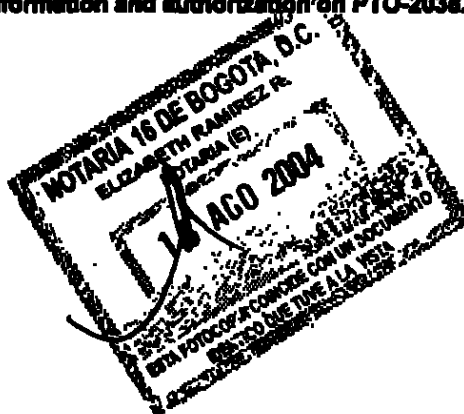
Docket Number

ACD 2916 PRUS

INVENTOR(S)/APPLICANT(S)		
Given Name (first and middle if any)	Family or Surname	Residence (City and either State or Foreign Country)
Koen Antoon Kornelis	Vanduffel	Deventer, Netherlands

Number 2 of 2

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Inventors:

- WESTMLIZE, Hans - Bathmen - NL
- SWIETEN VAN, Andreas Petrus - Velp - NL
- MEULENBRUGGE, Lambertus - Westervoort - NL
- 5 VANDUFFEL, Koen Antoon Kornelis - Deventer - NL

CONTINUOUS DOSING OF EXTREMELY FAST INITIATORS DURING POLYMERIZATION REACTIONS

10

The present invention relates to a process to polymerize one or more monomers by means of one or more organic initiators being dosed to the polymerization mixture at the reaction temperature.

15

Such a process is known from WO 00/17245, wherein initiators were dosed to a polymerization reactor at temperatures where the initiators have a half-life of from 0.05 to 1.0 hour. It is noted that the lower limit of this range (presenting the most reactive initiators) reflects the current opinion of the skilled person in the field that one cannot use a too reactive initiator for safety and efficiency reasons. More

20

specifically, hitherto it has always been expected that initiators with a half-life of 0.05 hour or less at conventional polymerization temperatures (especially for initiators used at temperatures from 40 to 70°C) could not be handled and transported safely because of inherent instability of the products. Furthermore, these so-called "extremely fast initiators" were not expected to efficiently

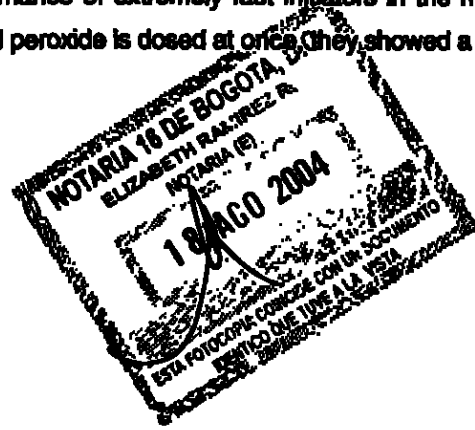
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polymerize monomers, since they were expected to give all types of side-reactions, such as hydrolysis, carboxy-inversion (for instance, wherein a peroxyester is converted to the corresponding carbonate, or a diacylperoxide to the corresponding anhydride), and other types of (in-cage) rearrangement reactions, leading to an unacceptably low initiation efficiency. Indeed, when

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evaluating the performance of extremely fast initiators in the more conventional processes wherein all peroxide is dosed at once, they showed a low efficiency.

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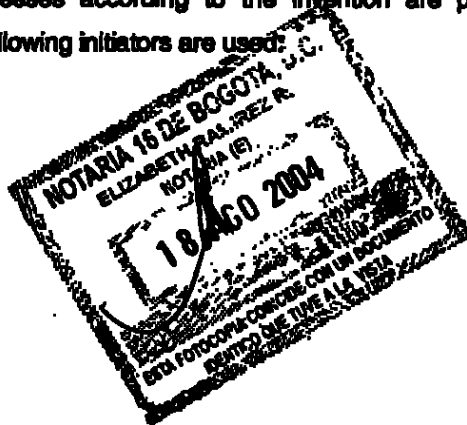
The current invention relates to a new process wherein extremely fast initiators are dosed to a reactor at polymerization conditions. This process gave an unexpected high initiator efficiency and allowed an even better control of the heat of polymerization being developed, allowing optimum reactor space-time yield, and even lower residual initiator levels in the resin after polymerization, when compared to the process and products of WO 00/17245. Accordingly, a resin with even improved heat stability properties and low fish-eye levels was obtained, while the polymerization time could be shortened.

10 The new process is characterized in that essentially all of the organic initiator used in the polymerization process has a half-life from 0.0001 hour to 0.0500 hour at the polymerization temperature, more preferably from 0.001 to 0.050 hour, most preferably from 0.002 to 0.050 hour.

15 The process according to the invention is pre-eminently suited to polymerize monomer mixtures comprising vinyl chloride monomer (VCM). Preferably, the process according to the invention involves the polymerization of monomer mixtures comprising at least 50% by weight (% w/w) of VCM, based on the weight of all monomer. Comonomers that can be used are of the conventional type and include vinylidene chloride, vinyl acetate, ethylene, propylene, acrylonitrile, styrene, and (meth)acrylates. More preferably, at least 80% w/w of the monomer(s) being polymerized is made up of VCM, while in the most preferred process the monomer consists essentially of VCM. As is known in the art, the polymerization temperature of such processes to a large extent determines the molecular weight of the resulting resin.

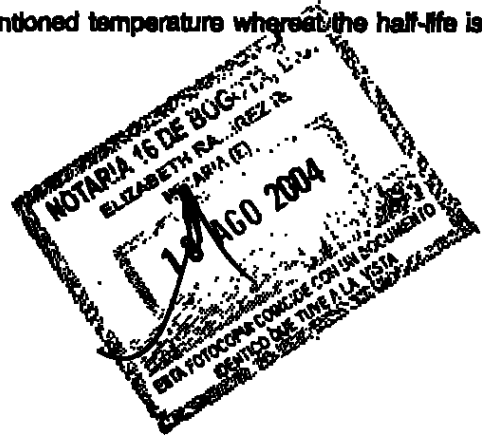
In the process according to the invention, one or more initiators may be used, as long as essentially all initiators that are used fulfill the half-life requirement.

30 Preferred examples of processes according to the invention are processes wherein one or more of the following initiators are used:



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- Hexanoyl pivaloyl peroxide, with a half-life of less than 0.05 hr at temperatures greater than 38°C,
 - 1,1,3,3-tetramethylbutylperoxy methoxy acetate, with a half-life of less than 0.05 hr at temperatures greater than 59°C,
 - 6 - 2,2-bis(2,2-dimethylpropanoylperoxy)-4-methyl pentane, with a half-life of less than 0.05 hr at temperatures greater than 61°C,
 - diisobutanoylperoxide, with a half-life of less than 0.05 hr at temperatures greater than 64°C,
 - bis(tert-butylperoxy) oxalate, with a half-life of less than 0.05 hr at temperatures greater than 67°C,
 - 10 - 2-(2,2-dimethylpropanoylperoxy)-2-(2-ethylhexanoylperoxy)-4-methyl pentane, with a half-life of less than 0.05 hr at temperatures greater than 69°C,
 - α -cumyl peroxyneodecanoate (Trigonox® 99), with a half-life of less than 0.05 hr at temperatures greater than 81°C,
 - 16 - 2,4,4-trimethylpentyl-2-peroxyneodecanoate, with a half-life of less than 0.05 hr at temperatures greater than 83°C,
 - peroxydicarbonates such as Trigonox® SBP, with a half-life of less than 0.05 hr at temperatures greater than 87°C,
 - 20 - peroxydicarbonates such as Perkadox® 16, with a half-life of less than 0.05 hr at temperatures greater than 88°C,
 - tert-amyl peroxyneodecanoate, with a half-life of less than 0.05 hr at temperatures greater than 88°C,
 - peroxydicarbonates such as Trigonox® EHP, with a half-life of less than 0.05 hr at temperatures greater than 89°C, and
 - 25 - tert-butyl peroxyneodecanoate, with a half-life of less than 0.05 hr at temperatures greater than 91°C,
- while they are dosed in a polymerization reaction mixture at or above the mentioned temperature whereat the half-life is 0.05°C, more preferably at a temperature from the mentioned temperature whereat the half-life is 0.05°C, up to 140°C.
- 30



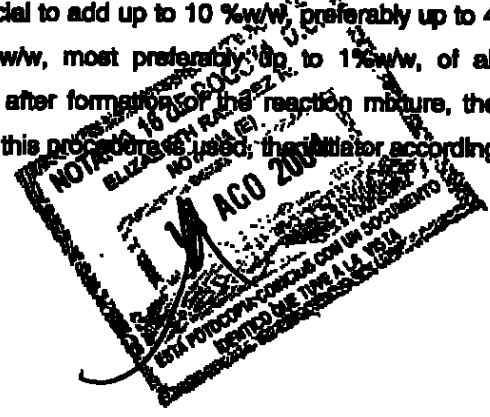
Other initiators may also be used. They can be classified on the basis of their half-lives as determined by conventional thermal decomposition studies in monochlorobenzene, as well-known in the art (see for instance the brochure
5 "Initiators for high polymers" with code 10737, obtainable from Akzo Nobel), and in accordance with the principles as set out above.

In this process it is not very efficient to use the extremely fast initiators during the polymerization stage wherein the content of the reactor is being heated-up to the
10 desired polymerization temperature, the so-called cold-start process. If so desired however, one or more of the extremely fast or more conventional initiators can be used at this stage.

It is not foreseen that one will use conventional initiators in any step of the
15 present process. However, if so desired, such conventional peroxides with a half-life of from 0.05 to 2 hours at the polymerization temperature, can be used in conjunction with the extremely fast initiators according to the invention. If a conventional initiator is used, it is advantageous to select it such that at the end of the polymerization the residual level is at an acceptably low level. Typically, this
20 will mean that if a conventional initiator is used, that this initiator will preferably be introduced in the reaction mixture prior to, or just after the heat-up step, when the conversion, or degree of polymerization, is below 80, preferably below 50, more preferably below 30, even more preferably below 10, and most preferably below 2 % of all monomer that is used in the process.

25 If the reaction mixture is formulated at or near the polymerization temperature, the so-called warm-start process, it is not required to add a certain amount of initiator at the start while the remainder is dosed over time. However, also in this warm-start process it can be beneficial to add up to 10 %w/w, preferably up to 4
30 %w/w, more preferably up to 2%w/w, most preferably up to 1%w/w, of all extremely fast initiator immediately after formation of the reaction mixture, the remainder being dosed over time. If this procedure is used, the initiator according

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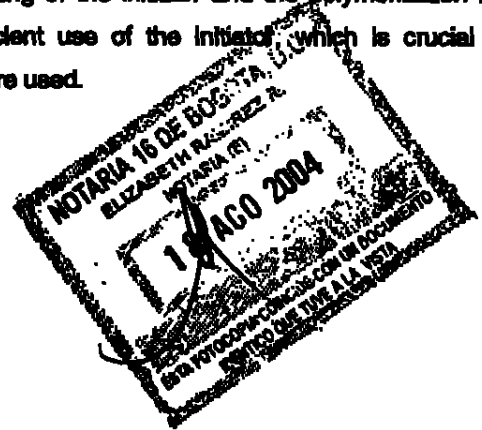


to the invention preferably is added as the last ingredient. This procedure is particularly preferred if a certain amount of polymerization inhibitor (a radical trapping species) is present in the reaction mixture. If such a radical scavenger is present, for instance because it is introduced with the monomer wherein it is typically used as a stabilizer, the initially dosed initiator will react with said scavenger, thus preventing a delayed start of the polymerization reaction.

The amount of initiator to be used in a process according to the invention is within the range conventionally used in polymerization processes. Typically, from 0.01 to 2 %w/w of initiator, preferably 0.01-1 %w/w, more preferably 0.01-0.5 %w/w, based on the weight of the monomer(s) to be polymerized, is used.

The initiator is dosed to the reactor in the pure form or, preferably, in the form of a dilute solution or dispersion (such as a suspension or emulsion). One or more suitable solvents can be used to dilute the initiator. Preferably, such solvents are easily removed during the steps working up the polymer after the polymerization process, such as alcohols, or they are of such a nature that it is acceptable to leave them as a residue in the final polymer, such as for solvents that are desired plasticizers for the final resin. Furthermore, it can be advantageous, but not necessarily required, that such solvents do not adversely affect the thermal stability of the initiator dissolved therein, as can be verified by analyzing the half-life temperature of the initiator in said solvent. An example of such a solvent is laododecane. If an initiator dispersion is dosed, then the dispersion can be of either the initiator itself or of a solution of the initiator, preferably in said suitable solvents. Preferably, the dispersion is an aqueous dispersion. Preferably, the initiator is dosed in a concentration of 0.1 to 50 %w/w, more preferably 0.5 to 25 %w/w, and most preferably 2 to 15 %w/w. The more dilute initiator solutions or dispersions ensure rapid mixing of the initiator and the polymerization mixture, which leads to a more efficient use of the initiator, which is crucial for the extremely fast initiators that are used.

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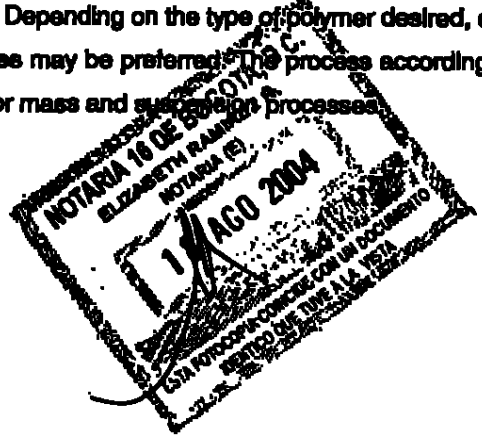


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The dosing of the extremely fast initiators to the reaction mixture can be intermittently or continuous over a period of time wherein at least 20%, preferably at least 40%, more preferably at least 60% of all monomer that is used in the process, is polymerized. If an Intermittent operation is selected there are at least 2, preferably at least 4, more preferably at least 10, and most preferably at least 20 moments at the polymerization temperature at which the initiator is dosed. If so desired, the intermittent and continuous operation can be combined, such that the initiator is dosed intermittently for certain (longer or shorter) periods of time. Most preferably, the peroxide is being dosed continuously and/or intermittently after that at least 5%, preferably at least 10%, more preferably at least 20%, most preferably at least 30% of the monomer(s) had already been polymerized and wherein during the dosing period at least 10%, preferably at least 20%, more preferably at least 30%, and most preferably at least 50% of all monomer that is used in the process, is polymerized.

Preferably, the dosing can be effected at any suitable entry point to the reactor. If water is added in the course of the polymerization process, for example to compensate for the shrinkage of the reactor content due to the polymerization reaction, it can be advantageous to use the line through which this water is dosed to also dose the initiator.

The polymerization process can be conducted either as a mass process wherein the reaction mixture is predominantly monomer or as a suspension process wherein the reaction mixture typically is a suspension of monomer in water, or as an emulsion or micro-emulsion process wherein the monomer typically is emulsified in water. In these processes the usual additives will have to be used. For example, if the monomer is present in the form of a suspension in water, the usual additives like surfactant(s), protective colloid(s), anti-fouling agent(s), pH-buffer(s), etc. may be present. Depending on the type of polymer desired, each of the above-mentioned processes may be preferred. The process according to the invention is especially suited for mass and suspension processes.

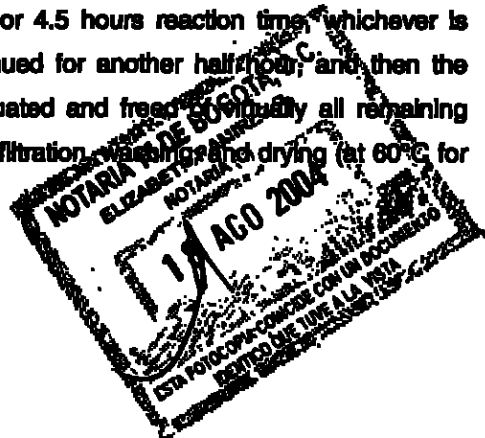


After the polymerization, the resulting (co)polymer (or resin) will be worked up as is usual in the art. Polymers obtained by a suspension polymerization according to the invention, for example, will be submitted to the usual drying and screening steps. The resulting resin preferably is characterized in that it contains less than 50 ppm of residual initiator, more preferably less than 40 ppm, and most preferably, less than 25 ppm of initiator, immediately after drying for 1 hour at 60°C and screening. The resin was found to exhibit excellent heat stability as measured with a MetraStat® PSD260 testing oven according to method ISO 182-2 (1990E). The improved heat stability proved that the resin hardly discolored when submitted to melt-processing steps, e.g., to form shaped articles.

To compare the existing procedure of warm start with the new proposed of the continuous peroxide dosing two thought experiments are carried out according to the below described experimental set-up

Experimental

In a standard suspension polymerization experiment, a temperature controlled 1-liter stainless steel Büchi reactor provided with one baffle, a three-bladed stirrer, a pressure transducer, a vinyl chloride (VCM) feed line, a nitrogen purge line, was charged with: 425 g demineralized water, 5 g of an 5% solution Alcotax® B72 (polyvinylacetate/alcohol) in demineralized water, and pressurized to 15 barg, using nitrogen. If no leaks are observed, the reactor is evacuated and pressurized with nitrogen up to 5 barg three times to flush out virtually all air. If not all initiator is dosed, then the desired amount of initiator is added. Next, the reactor was evacuated and charged with 250 g VCM ex Akzo Nobel Salt & Basics, followed by heating up of the reactor to polymerization temperature of 68°C in 30-60 min. After a pressure drop in the reactor, or 4.5 hours reaction time, whichever is shorter, the polymerization was continued for another half hour, and then the reactor was cooled to 20-25°C, evacuated and freed of virtually all remaining VCM. The polymer was obtained after filtration, washing and drying (at 60°C for 1 hour using a fluidized bed).



The initiator(s) used, the amount dosed, dosing procedure and the polymerization results are presented in the table 1.

The heat formed during polymerization process is measured and cooling adapted to the heat formed. The more heat is being generated at a certain time and the more difficult it will be to control the temperature of the reaction mixture. A low heat peak together with high polymer yields is preferred, since then an optimum in the space-time yield of the reactor can be attained.

Example 1

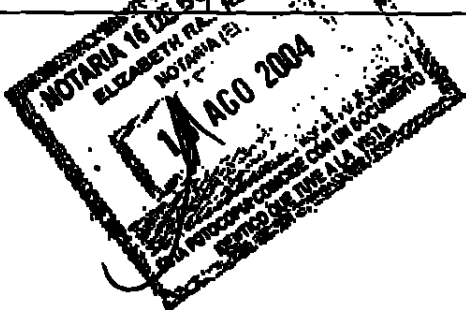
After reaching the polymerization temperature, 500 ppm of pure diisobutanoylperoxide (calculated on VCM), used as Trigonox® 187 at a concentration of 1% in laododecane, is dosed to the reaction mixture over a period of 30 minutes. Thereafter the dosing speed is reduced to 300 ppm of diisobutanoylperoxide per hour (using the same solution) over a period of 2.5 hrs. A pressure drop was observed. The results are mentioned in table 1

Comparative Example A

After reaching the polymerization temperature 1250 ppm of pure diisobutanoylperoxide (calculated on VCM), supplied as Trigonox® 187 in a 30% solution in laododecane, is added within two minutes. After 5 hours reaction time the polymerization is stopped and worked-up as described in the experimental procedure. The yield (based on computer simulation) is mentioned in table 1.

Table 1; Expected results of VCM suspension polymerization with Trigonox® 187 at 68°C according procedures for continuous dosing and for warm start.

	Example 1	Comparison
	Continuous dosing	Warm start
	500 ppm added during 30 min and subsequent 300 ppm at polymerization time	Peroxide added in 2 minutes at polymerization time

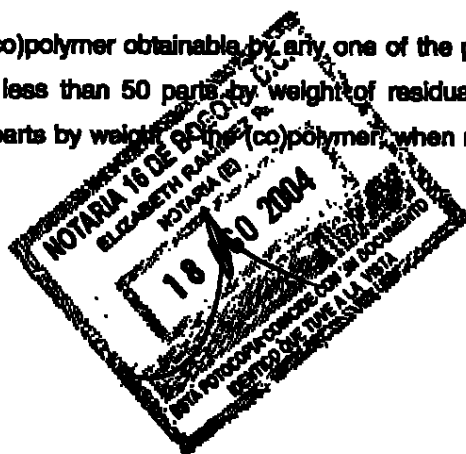


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Claims

1. Process to polymerize vinyl chloride monomer and optional further monomers using one or more organic initiators, with at least part of the initiator being dosed to the polymerization mixture at the reaction temperature, characterized in that essentially all of the organic initiator used in the polymerization process has a half-life of from 0.0001 hour to 0.05 hour at the polymerization temperature.
2. Process according to claim 1 wherein the initiator dosed at the reaction temperature is dosed continuously.
3. Process according to claim 1 or 2 wherein the dosing of the initiator is controlled such that the maximum cooling capacity of the reactor is used.
4. Process according to any one of claims 1-3 wherein the initiator, or part of the initiator, is dosed, intermittently and/or continuously, during a period wherein at least 20% of all monomer that is used in the polymerization, is polymerized.
5. Process according to any one of claims 1-3 wherein the initiator, or part of the initiator, is dosed, intermittently and/or continuously, after that at least 5%, preferably at least 10%, more preferably at least 20%, most preferably at least 30% of the monomer(s) had already been polymerized and wherein during the dosing period at least 10%, preferably at least 20%, more preferably at least 30%, and most preferably at least 50% of all monomer that is used in the process, is polymerized.
6. Vinyl chloride based (co)polymer obtainable by any one of the processes of claims 1-4, having less than 50 parts by weight of residual initiator, based on one million parts by weight of (co)polymer, when measured

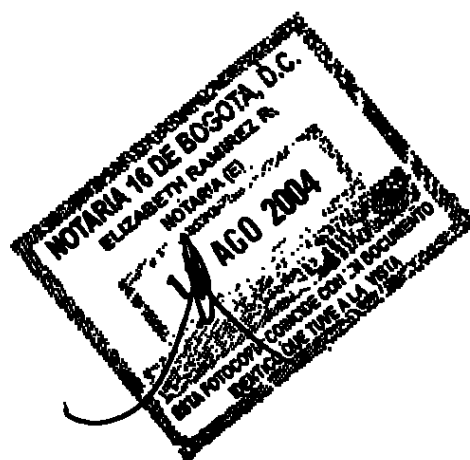


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Immediately after polymerization and drying of the (co)polymer for 1 hour at 60°C.

- 5 7. Use of a vinyl chloride (co)polymer according to claim 5 in a shaping process involving the heating of the co(polymer) to above its melting temperature.

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Abstract

The invention pertains to a process wherein initiators are dosed to a polymerization mixture, with essentially all of the organic initiator that is used in the polymerization process having a half-life from 0.0001 hour to 0.05 hour at the polymerization temperature. The dosing of such initiator allows for an improved control of the polymerization rate, and the process will render a polymer with very low residual initiator levels.



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Bogotá D C

Doctor(a)
EMILIO FERRERO.
Apoderado(a)

REFERENCIA:	Radicación N°	04-56932
	Solicitud internacional	PCT/EP02/14371
	Fecha límite de inicio en fase nacional	21/07/2004
	Trámite	011
	Evento	379
	Actuación	416
	Oficio N°	1039
	Folios	001

Teniendo en cuenta que la solicitud de privilegio de patente que se tramita bajo el expediente indicado en el asunto, reúne los requisitos de forma establecidos en los artículos 26 y 27 de la Decisión 486, de la Comisión de la Comunidad Andina, el Tratado de Cooperación en Materia de Patentes (PCT), el Reglamento y la Circular Única de la Superintendencia de Industria y comercio, 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 dispone el artículo 40 de la referida Decisión 486.

Cúmplase
Dado en Bogotá D C,

19/10 OCT. 2005


ALIX CARMENZA CESPEDES DE VERGEL
Jefe División de Nuevas Creaciones.

202028

Al contestar favor indique el número de radicación consignado en el sticker

Sede Centro: Carrera 13 No. 27-00 Pisos 2, 5, 7 y 10
Commutador: 3 820840 Fax: 3 505220
Sede CAN: Tr. 40A No. 38-50 31 53265 al 69
Fax: 3 505220. Línea 9800-910 165
Web: www.sic.gov.co e-mail: info@sic.gov.co
Bogotá D.C. - Colombia