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Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a),(b) or (b-bis)

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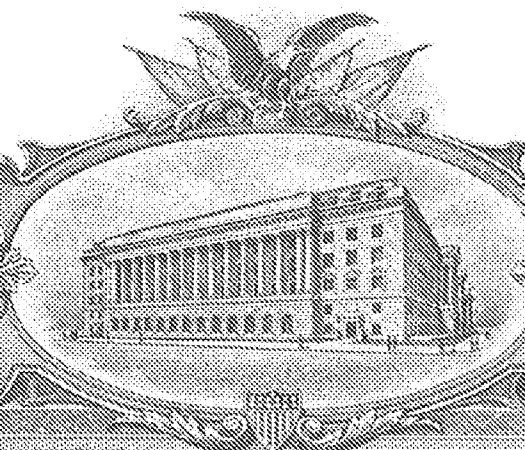
Application number: 63332443

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THE UNITED STATES OF AMERICA

TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE

United States Patent and Trademark Office

May 22, 2023

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

APPLICATION NUMBER: 63/332,443
FILING DATE: *April 19, 2022*

THE COUNTRY CODE AND NUMBER OF YOUR PRIORITY APPLICATION, TO BE USED FOR FILING ABROAD UNDER THE PARIS CONVENTION, IS US63/332,443



Certified by

Kathi

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

PROVISIONAL APPLICATION FOR PATENT COVER SHEET

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

INVENTOR(S)

Given Name (first and middle (if any))	Family Name or Surname	Residence (City and either State or Foreign Country)
Harry	CHMIELEWSKI	Jacksonville, Florida
Tina	MURGUIA	Jacksonville, Florida
Mike	PEACH	Jacksonville, Florida
Priyanka	BHATTACHARYA	Jacksonville, Florida
Susan	SHINALL	Jacksonville, Florida
Larissa	FENN	Jacksonville, Florida

☐ Additional inventors are being named on the separately numbered sheets attached hereto

TITLE OF THE INVENTION (280 characters max)

SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS

CORRESPONDENCE ADDRESS

Direct all correspondence to:

☐ Customer Number

_____ *Type Customer Number here*

-

_____ *Place Customer Number Bar Code Label here*

OR

CORRESPONDENCE ADDRESS

CUSTOMER NO.
00513

/Peter Jay/ 2022.04.19
11:37:23 -04'00'

By: _____

Peter M. Jay
Registration No. 66,363

WENDEROTH, LIND & PONACK, L.L.P.
1025 Connecticut Avenue, N.W., Suite 500
Washington, D.C. 20036
Phone: (202) 721-8200
Fax: (202) 721-8250
April 19, 2022

ENCLOSED APPLICATION PARTS (check all that apply)

☒ Specification *Number of Pages* [49] ☐ CD(s), Number _____
☒ Drawings *Number of Sheets* [2] ☒ Other, (specify) Application Data Sheet

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT (check one)

- ☐ Applicant claims small entity status. See 37 CFR 1.27.
☐ A check or money order is enclosed to cover the filing fees of \$ 300.00
☒ The Commissioner is hereby authorized to charge filing fees or credit any overpayment to
Deposit Account No. 23-0975. Filing Fee Amount \$300.00
☒ Please charge my Credit Card in the amount of \$300.00 to cover the above fees.

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: _____

The USPTO is hereby authorized to charge any fees under 37 C.F.R. §§ 1.16, 1.17, and 1.492, which may be required by this paper and for the entire pendency of the present application to Deposit Account No. 23-0975, except fees for multiple dependent claims.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	PR22-0095A
		Application Number	
Title of Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS		
The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76. This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.			

Secrecy Order 37 CFR 5.2:

☐	Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)
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Inventor Information:

Inventor	1				Remove	
Legal Name						
Prefix	Given Name	Middle Name	Family Name	Suffix		
	Harry		CHMIELEWSKI			
Residence Information (Select One) • US Residency Non US Residency Active US Military Service						
City	Jacksonville	State/Province	FL	Country of Residence	US	
Mailing Address of Inventor:						
Address 1	c/o Rayonier Advanced Materials Inc.					
Address 2	1301 Riverplace Blvd., Suite 2300					
City	Jacksonville	State/Province	FL			
Postal Code	32207	Country i	US			
Inventor	2				Remove	
Legal Name						
Prefix	Given Name	Middle Name	Family Name	Suffix		
	Tina		MURGUIA			
Residence Information (Select One) • US Residency Non US Residency Active US Military Service						
City	Jacksonville	State/Province	FL	Country of Residence	US	
Mailing Address of Inventor:						
Address 1	c/o Rayonier Advanced Materials Inc.					
Address 2	1301 Riverplace Blvd., Suite 2300					
City	Jacksonville	State/Province	FL			
Postal Code	32207	Country i	US			
Inventor	3				Remove	
Legal Name						
Prefix	Given Name	Middle Name	Family Name	Suffix		
	Mike		PEACH			
Residence Information (Select One) • US Residency Non US Residency Active US Military Service						

Application Data Sheet 37 CFR 1.76		Attorney Docket Number		PR22-0095A	
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City	Jacksonville	State/Province	FL	Country of Residence ⁱ	US
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Mailing Address of Inventor:

Address 1	c/o Rayonier Advanced Materials Inc.				
Address 2	1301 Riverplace Blvd., Suite 2300				
City	Jacksonville	State/Province	FL	Country ⁱ	US
Postal Code	32207	Country ⁱ	US		
Inventor	4				Remove
Legal Name					
Prefix	Given Name	Middle Name	Family Name	Suffix	
	Priyanka		BHATTACHARYA		
Residence Information (Select One) ☒ US Residency ☐ Non US Residency ☐ Active US Military Service					
City	Jacksonville	State/Province	FL	Country of Residence ⁱ	US

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City	Jacksonville	State/Province	FL	Country ⁱ	US
Postal Code	32207	Country ⁱ	US		
Inventor	5				Remove
Legal Name					
Prefix	Given Name	Middle Name	Family Name	Suffix	
	Susan		SHINALL		
Residence Information (Select One) ☒ US Residency ☐ Non US Residency ☐ Active US Military Service					
City	Jacksonville	State/Province	FL	Country of Residence ⁱ	US

Mailing Address of Inventor:

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Address 2	1301 Riverplace Blvd., Suite 2300				
City	Jacksonville	State/Province	FL	Country ⁱ	US
Postal Code	32207	Country ⁱ	US		
Inventor	6				Remove
Legal Name					
Prefix	Given Name	Middle Name	Family Name	Suffix	
	Larissa		FENN		
Residence Information (Select One) ☒ US Residency ☐ Non US Residency ☐ Active US Military Service					
City	Jacksonville	State/Province	FL	Country of Residence ⁱ	US

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number		PR22-0095A	
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Address 1		c/o Rayonier Advanced Materials Inc.			
Address 2		1301 Riverplace Blvd., Suite 2300			
City	Jacksonville	State/Province		FL	
Postal Code	32207	Country	US		
All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the Add button. Add					

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below. For further information see 37 CFR 1.33(a).			
☐ An Address is being provided for the correspondence information of this application.			
Customer Number	00513		
Email Address	wlp@wenderoth.com	Add Email	Remove Email

Application Information:

Title of the Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS		
Attorney Docket Number	PR22-0095A	Small Entity Status Claimed ☐	
Application Type	Provisional		
Subject Matter	Utility		
Total Number of Drawing Sheets (if any)	2	Suggested Figure for Publication (if any)	

Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country

Publication Information:

☐ Request Early Publication (Fee required at time of Request 37 CFR 1.219)
☐ Request Not to Publish. I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application has not and will not be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	PR22-0095A
		Application Number	
Title of Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS		

Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32). Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

Please Select One:	☒ Customer Number	☐ US Patent Practitioner	☐ Limited Recognition (37 CFR 11.9)
Customer Number	00513		

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78. When referring to the current application, please leave the "Application Number" field blank.

Prior Application Status		Remove
Application Number	Continuity Type	Prior Application Number
Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the Add button.		Add

Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)ⁱ the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

Application Number	Country ⁱ	Filing Date (YYYY-MM-DD)	Remove
			Access Code ^j (if applicable)
Additional Foreign Priority Data may be generated within this form by selecting the Add button.			Add

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	PR22-0095A
		Application Number	
Title of Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS		

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013. ☐ NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.	
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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	PR22-0095A
		Application Number	
Title of Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS		

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	PR22-0095A
		Application Number	
Title of Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS		

Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Applicant	1	Remove
If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section. Clear		
☒ Assignee	Legal Representative under 35 U.S.C. 117	Joint Inventor
Person to whom the inventor is obligated to assign.		Person who shows sufficient proprietary interest
If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:		
Name of the Deceased or Legally Incapacitated Inventor:		
If the Applicant is an Organization check here. ☒		
Organization Name	Rayonier Advanced Materials Inc.	
Mailing Address Information For Applicant:		
Address 1	1301 Riverplace Blvd., Suite 2300	
Address 2		
City	Jacksonville	State/Province FL
Country	US	Postal Code 32207
Phone Number		Fax Number
Email Address		
Additional Applicant Data may be generated within this form by selecting the Add button. Add		

Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76	Attorney Docket Number	PR22-0095A
	Application Number	
Title of Invention	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS	

Assignee	1		
Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.			
			Remove
If the Assignee or Non-Applicant Assignee is an Organization check here.			☒
Organization Name	Rayonier Advanced Materials Inc.		
Mailing Address Information For Assignee including Non-Applicant Assignee:			
Address 1	1301 Riverplace Blvd., Suite 2300		
Address 2			
City	Jacksonville	State/Province	FL
Country i	US	Postal Code	32207
Phone Number		Fax Number	
Email Address			
Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button.			Add

Signature:[Remove](#)

NOTE: This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). **However, if this Application Data Sheet is submitted with the INITIAL filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).**

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

Signature	/Peter M. Jay/		Date (YYYY-MM-DD)	2022-04-19	
First Name	Peter	Last Name	Jay	Registration Number	66363
Additional Signature may be generated within this form by selecting the Add button.				Add	

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Application Data Sheet 37 CFR 1.76		Attorney Docket Number	PR22-0095A
		Application Number	
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This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

TITLE: SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS
FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT
PRODUCTS

TECHNICAL FIELD

5 [0001] The present disclosure relates to solutions containing cyclodextrins for imparting odor control to materials used in absorbent products, substrates treated with solutions containing cyclodextrins for imparting odor control to materials used in absorbent products, and associated products and methods.

BACKGROUND

10 [0002] Absorbent products, such as adult incontinence liners, bladder control pads, diapers, pull-up underwear, etc., should eliminate the odor of fresh urine in a short amount of time. Such products are often single-use products that are worn in public. Their users value odor protection to avoid any possibility of embarrassment. Even though many commercially available products claim to provide some type of odor control, these commercially available products can be
15 ineffective at controlling the odor of fresh urine at the moment of use of the product. The odor of fresh urine at body temperature can often be easily detected on these absorbent products. The urine of older individuals can be particularly noticeable. Therefore, the industry still desires more effective odor control, especially in absorbent products.

[0003] Many have attempted to solve the problems associated with controlling odors in
20 absorbent products. In some of the main approaches, odor control involves inhibiting the growth of odor-causing bacteria and adsorbing odoriferous molecules on high-surface-area adsorbents. However, such conventional technologies are generally too slow and/or ineffective at reducing the odor of fresh urine in a short amount of time.

[0004] In another approach, cyclodextrins are used for odor control in absorbent products. For
25 instance, U.S. Pat. No. 5,429,628 discloses the use of free, uncomplexed cyclodextrins to effectively absorb malodor. U.S. Pat. No. 5,429,628 uses small particle size cyclodextrin to increase the surface area available to absorb odoriferous molecules, and thus improve odor

absorption efficacy. U.S. Pat. No. 5,429,628 also teaches perfume/cyclodextrin complexes formed either by bringing the perfume and cyclodextrin together in a suitable solvent, such as water, or, preferably, by kneading and/or slurring the ingredients together in the presence of a minimal amount of solvent, preferably water. The kneading/slurring method is particularly desirable because the method produces smaller solid particle sizes. After freeze-drying or spray drying, the solid complexes can be ground into particles that have a particle size of less than about 10 μm . However, care must be given when grinding so as to not generate a workplace safety hazard by with respirable particles.

[0005] Further, the use of aqueous odor-absorbing solutions for use on odoriferous surfaces has also been described, for example, in U.S. Pat. No. 6,284,231. The composition of solutions disclosed in U.S. Pat. No. 6,284,231 comprises from about 0.1% to about 20% of a solubilized, water-soluble, uncomplexed cyclodextrin, an effective amount of at least one ingredient to improve the odor-absorbing performance of the cyclodextrin and a water-soluble antimicrobial active. These solutions are typically provided in a spray dispenser and have been commercialized under the trade name FEBREZE®.

[0006] Even though conventional technology provides cyclodextrins that can generally be used to absorb odors, it has not been straightforward to incorporate fine, dry particles of a cyclodextrin complex into an absorbent article. The dry complex powder can be sprinkled, mixed, or distributed onto the absorbent product. However, in practice, these methods of application have not been satisfactory. The immobilization of a cyclodextrin/perfume complex should not involve an excessive amount of water to avoid premature release of the perfume. Water-soluble binders, e.g., a low melting temperature polymeric binder such as polyethylene glycol, have been disclosed. Another disclosed method is the application of a cyclodextrin/perfume slurry directly to the absorbent product with a drying step. However, in practice, these slurries are difficult to handle, dispense, and uniformly apply.

[0007] To create a cyclodextrin complex in the form of a low viscosity liquid that can be more easily handled during the manufacturing of absorbent articles, and to avoid settling of insoluble cyclodextrin particles, U.S. Pat. No. 9,592,168 teaches an absorbent article comprising an inclusion complex of cyclodextrin with an organic compound made using colloidal milling and

spray drying processes, whereby the inclusion complex is dispersed in a matrix comprising polysiloxane oil. The creation of dry particles of colloidal dimensions allowed for the production of a homogeneous dispersion of solid particles that was used in the preparation of absorbent articles.

- 5 [0008] U.S. Pat. No. 5,429,628 provides examples of applying cyclodextrin to Kraft cellulose fibers by dissolving cyclodextrin in water at elevated temperature to form a clear solution and spraying the solution onto the fibers. All of the examples require drying of the water carrier from the substrate either by air or oven drying or freeze-drying. The low solubility of the cyclodextrin requires a high solution add-on to achieve an effective level of performance, and the
- 10 high water content of the solution requires drying of the cellulose fiber to prevent microbial growth during storage. Fluff pulp typically contains 6-8% water after drying. Microbial growth can occur if the water content increases into a range of about 13-15%. Therefore, all of these examples are impractical without an additional drying step.

- [0009] U.S. Pat. No. 5,571,782 similarly teaches the use of an effective amount of
- 15 cyclodextrin/active complexes in the form of solid particles having particle sizes below about 12 μm for fast release of the perfume active when wetted. Handling of these solid particles is straightforward in fabric softener and solid detergent compositions but can be awkward and problematic when attempting to apply the solid particles to absorbent materials and/or absorbent products.

- 20 [0010] US Pat. Nos. 10,183,273 and 10,427,133 claim layers of an absorbent article treated with a substituted cyclodextrin complex (i.e., a degree of substitution = 0.4 to 2.5 R per glucose unit of cyclodextrin) and an odor-controlling organic compound in powder form or dispersed in a carrier to form a dispersion for application to an absorbent article. The carrier was polysiloxane oil, mineral oil, petrolatum, polyethylene glycol, and/or glycerine. US Pat. Nos. 10,183,273 and
- 25 10,427,133 also describe methods of application using cyclodextrin and an odor-controlling organic compound in a volatile solvent system containing at least 60% water, alcohols, ketones or aldehydes, hydrocarbons, or supercritical fluids. In some forms, the solution is easily pumpable or sprayable directly onto an absorbent article. However, this approach used only substituted cyclodextrins, which are believed to stack upon crystallization into a physical form

that is superior to unsubstituted cyclodextrins for the rapid release of the odor-controlling compound complexed with the cyclodextrin.

[0011] U.S. Pat. No. 8,574,683 teaches a method of making a pulp sheet of odor-inhibiting absorbent fibers comprising a cyclodextrin odor absorbent and a biocide dissolved in a non-water liquid carrier comprised of less than 20% water and applied to a pulp sheet of cellulose fibers. Many of the perfumes and essential oils included in the present disclosure, such as citral, have been shown to have biocidal activity, including in, for example, the following disclosures: K. Winska, et. al. "Essential Oils as Antimicrobial Agents—Myth or Real Alternative?" *Molecules* (2019) Jun; 24(11): 2130, C. Shi. et. al. "Antimicrobial Activity and Possible Mechanism of Action of Citral against *Cronobacter sakazakii*" *PLoS One* 2016; 11(7), and M, Choudhary, et. al. "*Lemongrass Essential Oil Components with Antimicrobial and Anticancer Activities*" *Antioxidants* 2021 Dec 22; 11(1): 20.

[0012] Even though the above-referenced applications and conventional technologies can be used to reduce odors, the need still exists for a convenient way of controlling the odor of fresh urine in absorbent products at the moment of use. That is, many of the prior approaches to odor control act too slowly for reducing the odor of fresh urine in a product at the moment of use and/or require additional processing that imparts additional costs. The inventors solved the problems with conventional approaches and provide solutions that are effective at reducing the odor of fresh urine in a very short amount of time. Users of these products value odor protection to avoid any chance of embarrassment. The present disclosure, as discussed in detail below, provides ways to achieve these and other objectives by making solutions containing relatively high concentrations of cyclodextrin complexes that can be easily spray applied to materials used in absorbent products without requiring a drying step. The moisture increase of a treated absorbent material should be <5% by weight or even <1% by weight when a drying step is not used.

BRIEF SUMMARY

[0013] In view of the above-mentioned exemplary problems with conventional and known cyclodextrin applications, the present application provides new and improved solutions containing cyclodextrins for imparting odor control to materials used in absorbent products.

5 [0014] An embodiment of the present application includes a substrate comprising an odor-controlling solution applied thereupon, wherein the odor-controlling solution comprises: a polar organic solvent, water, a cyclodextrin, and an odor-controlling compound, and wherein the substrate comprises at least 0.2 kg of the odor controlling compound per 1000 kg of the substrate.

10 [0015] In one embodiment, the substrate has an increase in moisture content of less than 1 wt. % as a result of the odor controlling solution having been applied thereupon.

[0016] In another embodiment, the substrate is a fluff pulp in the form of a compressed sheet or an air-laid fiber form, a synthetic or cellulosic fiber spun-bond or carded nonwoven, woven fabric, or solid material, or an absorbent article.

15 [0017] In another embodiment, the substrate is the solid material, and wherein the solid material is selected from the group consisting of a polymeric foam, leather, and plastic film.

[0018] In another embodiment, the substrate material is the absorbent article, and wherein the absorbent article is an adult incontinence control pad, feminine hygiene pad, pull-up underwear, adult brief, baby diaper, or bed pad.

20 [0019] In another embodiment, the polar organic solvent is selected from the group consisting of glycerol, propylene glycol, polypropylene glycol, polyethylene glycol, and mixtures thereof.

[0020] In another embodiment, the cyclodextrin is an unsubstituted or substituted α -, β - or γ -cyclodextrin.

25 [0021] In another embodiment, the odor controlling compound is at least one of an essential oil selected from the group consisting of citral, limonene, lavandin, geranium, grapefruit, linalool,

citronellol, linalyl acetate, geraniol, eugenol, floracetate, cardamom, eucalyptus, juniper berry, lavender, lemon verbena, lemongrass, patchouli, rose, spearmint, tea tree, oregano, persimmon and mixtures thereof, a botanical extract, an odor-controlling substance selected from the group consisting of benzaldehyde, cinnamic aldehyde, ligustral, florhydral, and mixtures thereof, or an antimicrobial substance selected from the group consisting eucalyptus, lavender, thyme, peppermint, cajuput, cinnamon, clove, sage, tea tree oils, and mixture thereof.

[0022] In another embodiment, the odor controlling solution comprises 1.5 to 1.75 moles of the odor controlling compound per mole of the cyclodextrin.

[0023] In another embodiment, the odor-controlling solution has a pH greater than or equal to 12.0.

[0024] In another embodiment, the odor controlling solution comprises 67 wt. % of glycerol as the polar organic solvent, 12 wt. % of the water, and 21 wt. % of the cyclodextrin and the odor-controlling compound, wherein the cyclodextrin is unsubstituted β -cyclodextrin, and wherein the odor-controlling compound is citral, wherein the citral and the unsubstituted β -cyclodextrin are present in a mole ratio of 1.5 moles of the citral per mole of the unsubstituted β -cyclodextrin, wherein the substrate is fluff pulp, wherein the substrate comprises 0.4 kg of the citral per 1000 kg of the fluff pulp, and wherein the substrate has an increase in moisture content of less than 0.13 wt. % as a result of the odor controlling solution having been applied thereupon.

[0025] Another embodiment of the present application includes an article comprising the substrate described above.

[0026] Yet another embodiment of the present application includes a method of preparing an odor-controlling substrate, comprising: a) providing a substrate, b) applying an odor-controlling solution onto the substrate to obtain the odor-controlling substrate, wherein the odor-controlling solution comprises: a polar organic solvent, water, a cyclodextrin, and an odor-controlling compound, and wherein, after step b), the odor controlling substrate comprises at least 0.2 kg of the odor controlling compound per 1000 kg of the odor controlling substrate.

[0027] In another embodiment, applying the odor-controlling solution onto the substrate in aforementioned step b) increases moisture content of the substrate by less than 1 wt. %.

[0028] In another embodiment, the substrate is a fluff pulp in the form of a compressed sheet or an air-laid fiber form, a synthetic or cellulosic fiber spun-bond or carded nonwoven, woven
5 fabric, or solid material, or an absorbent article.

[0029] In another embodiment, the polar organic solvent is selected from the group consisting of glycerol, propylene glycol, polypropylene glycol, polyethylene glycol, and mixtures thereof.

[0030] In another embodiment, the cyclodextrin is an unsubstituted or substituted α -, β - or γ -cyclodextrin.

10 [0031] In another embodiment, the odor controlling solution comprises 1.5 to 1.75 moles of the odor controlling compound per mole of the cyclodextrin.

[0032] In another embodiment, the odor-controlling solution has a pH greater than or equal to 12.0.

15 [0033] In another embodiment, the odor controlling solution comprises 67 wt. % of glycerol as the polar organic solvent, 12 wt. % of the water, and 21 wt. % of the cyclodextrin and the odor-controlling compound, wherein the cyclodextrin is unsubstituted β -cyclodextrin, and wherein the odor-controlling compound is citral, wherein the citral and the unsubstituted β -cyclodextrin are present in a mole ratio of 1.5 moles of the citral per mole of the unsubstituted β -cyclodextrin, wherein the substrate is fluff pulp, wherein the substrate comprises 0.4 kg of the citral per 1000
20 kg of the fluff pulp, and wherein the substrate has an increase in moisture content of less than 0.13 wt. % as a result of the odor controlling solution having been applied thereupon.

[0034] In another embodiment, step b) comprises applying 0.011 g of the odor-controlling solution per g of the substrate material at a solution temperature of 86° C and pH 12.5, wherein the substrate material is a sheet of fluff pulp.

25 [0035] Yet another embodiment of the present application includes a substrate comprising an odor-controlling solution applied thereupon, wherein the odor-controlling solution comprises:

water, an optional polar organic solvent, a cyclodextrin, and an odor-controlling compound, and wherein the substrate comprises at least 0.2 kg of the odor-controlling compound per 1000 kg of the substrate, and wherein the odor-controlling solution comprises 1.5 to 1.75 moles of the odor-controlling compound per mole of the cyclodextrin, and wherein the odor-controlling solution
5 has a pH greater than pH 12.0, and wherein the concentration of cyclodextrin and odor-control compound in the solution is about 18 wt%, and wherein the substrate has an increase in moisture content of about 1.1 wt% as a result of the odor controlling solution having been applied thereupon.

[0036] Other objects, features, and advantages of the present invention will be apparent to those
10 of ordinary skill in the art in view of the following detailed description of the invention and accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

[0037] The drawings as provided herein set forth some exemplary embodiments of the solutions containing cyclodextrins for imparting odor control to materials used in absorbent products of
15 the present application, the detailed description of which follows. The drawings are merely exemplary and are not intended to limit the invention.

[0038] FIG. 1 is a chart showing the dose and response of citral for urine odor control.

[0039] FIG. 2 is a photomicrograph at 20X with 100 μm for the smallest division, which shows particles of citral / β -cyclodextrin complexes precipitated from 86° C glycerol/water solutions at
20 pH 12.5.

[0040] FIG. 3 is a photomicrograph at 20X with 100 μm for the smallest division, which shows particles of citral / β -cyclodextrin complexes precipitated from 86° C glycerol/water solutions at pH 9.6.

DETAILED DESCRIPTION

[0041] The solutions containing cyclodextrins for imparting odor control to materials used in absorbent products, and associated products and methods of the present application are now
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described by reference to the embodiments. The description provided herein is not intended to limit the scope of the claims, but to exemplify the variety encompassed by the present application. The embodiments are described more fully hereinafter with reference to the accompanying drawings in which like numerals represent like elements throughout the several
5 figures, and in which example embodiments are shown. Embodiments of the claims may, however, be embodied in many different forms and should not be construed as limited to the embodiments set forth herein. The examples set forth herein are non-limiting examples and are merely examples among other possible examples.

[0042] As discussed above, the need exists for technologies for absorbent products, such as adult
10 incontinence liners, bladder control pads, and pull-up underwear, to eliminate the odor of fresh urine at short times. Since such products are often single-use products that are worn in public, users value odor protection to avoid any possibility of embarrassment. Even though many commercially available products claim some type of odor control, these products can be ineffective at controlling the odor of fresh urine at the moment of use of the product. The odor
15 of fresh urine at body temperature can be easily detected on these absorbent products.

[0043] To solve the problems with conventional technology, the present disclosure uses rapid-acting odor-masking and odor-control agents that can be incorporated into a cyclodextrin complex and deposited on fluff fibers. The active ingredients are stabilized in the complex for long shelf life and released only when the fibers contact urine. Many essential oil fragrances and
20 organic odor-control agents can be used to make stable cyclodextrin complexes that are highly soluble in polar organic and water solvent systems. These soluble complexes can be sprayed uniformly or otherwise applied to sheets of fluff fibers. *In situ* precipitation of the complex on the fiber surface after cooling to room temperature provides unexpectedly efficient use of the odor-masking agents and delivers excellent odor control when the fibers become hydrated with
25 urine. Thus, the present disclosure relates to a novel solution applied at a specific add-on amount having: (1) an optimal essential oil to cyclodextrin molar ratio and solution pH; (2) a sufficient concentration of a soluble cyclodextrin complex to impart urine odor control when applied to a material used in an absorbent product; and (3) a concentration of water low enough so as not to require an additional drying step after its application.

[0044] Cyclodextrins are cyclic oligosaccharides containing six to twelve glucose units that can form inclusion complexes with host molecules that can fit wholly or partially within the cyclodextrin cavity. Inclusion complexes comprised of highly volatile essential oils and other odor control ingredients can be deposited on the surface of a substrate in a stable dry form. A stable, dry form is needed because volatile essential oils are a costly component of the formulation and should not evaporate from a treated substrate. The host molecules, especially the scent of an essential oil, can be released by the action of water and urine. The present disclosure relates to a solution whose composition, pH, and temperature are favorable for making a soluble complex comprised of cyclodextrin and an essential oil or odor control compound, such that the solution containing the soluble complex can be sprayed or otherwise applied to a material used in an absorbent product, such that the complex precipitates *in situ* on the surface of the material upon cooling to room temperature, and the material treated with the complex reduces the odor of body fluids such as urine, menses, and sweat.

[0045] Cyclodextrin complexes containing a cyclodextrin and odor-controlling compounds tend to have low water solubility and can be difficult to uniformly apply and be retained on the surface of a solid fiber or film. Solutions containing a polar organic solvent and water at an elevated pH and temperature provide the capability to form a cyclodextrin complex in solution and also to provide the solubility required for uniform application of a homogeneous solution of a cyclodextrin complex onto a solid surface. Upon cooling to room temperature, the cyclodextrin complex precipitates *in situ* on the solid fiber surface. Because the cyclodextrin complex precipitates *in situ* on the solid fiber surface, the control of particle sizes is also not required. Typically, complexes are formed by mixing or kneading an odor control compound and cyclodextrin together in the presence of a small amount of water. After freeze-drying or spray drying, the complexes are ground to a particle size less than about 10 μm . Such a process is not needed due to the *in situ* precipitation utilized by the presently disclosed solutions. Additionally, colloid stabilization for making and applying complex is not required. Therefore, cyclodextrin complexes can be easily prepared and applied to absorbent materials and cellulosic materials, even without requiring a drying step.

[0046] Solutions Containing Cyclodextrins For Imparting Odor Control

[0047] The odor-controlling solutions of the present disclosure comprise an optional polar organic solvent, water, a cyclodextrin, and an odor-controlling compound.

[0048] The polar organic solvent can be any polar organic solvent capable of providing the capability to form a cyclodextrin complex in solution and also to provide the solubility required for uniform application of a homogeneous solution of a cyclodextrin complex onto a solid surface. Examples of acceptable polar organic solvents include, but are not limited to, glycerol, propylene glycol, polypropylene glycol, polyethylene glycol, and mixtures thereof. In particular, glycerol can be used as a polar organic solvent. An increase in the amount of the polar organic solvent in the solution can increase the solubility of a cyclodextrin complex in the solution. For instance, the solubility of the complex can be increased so that an effective amount of complex can be applied to a substrate without an excessive increase in moisture. TABLE 1 below shows the increase in substrate moisture for two levels of add-on (i.e., 0.4 kg of essential oil /ADMT fluff pulp and 2.0 kg of essential oil /ADMT fluff pulp) as a function of glycerol concentration.

15 [0049] [TABLE 1]

	Target Citral Add-On (kg Citral/ADMT fluff)	Citral Loading (mol Citral/mol CD)	Total Solids Add-On (g solids /g fluff)	Liquid Composition (fraction glycerol)	Liquid Composition (fraction water)	Solution Solubility (g total solids/ g solution)	Solution Solubility (g CD/ g solution)	Solution Add-on (g soln /g fluff)	Fluff Moisture Increase (%)	Glycerol Add-On (kg glycerol /ADMT)	CD Add-On (kg CD /ADMT)
1	0.4	1.50	0.0024	0.85	0.15	0.197	0.164	0.012	0.1%	8.3	2.0
2	0.4	1.50	0.0024	0.50	0.50	0.197	0.164	0.012	0.5%	4.9	2.0
3	0.4	1.50	0.0024	0.00	1.00	0.197	0.164	0.012	1.0%	0.0	2.0
4	2.0	1.50	0.0119	0.85	0.15	0.197	0.164	0.061	0.7%	41.3	9.9
5	2.0	1.50	0.0119	0.50	0.50	0.197	0.164	0.061	2.4%	24.3	9.9
6	2.0	1.50	0.0119	0.00	1.00	0.197	0.164	0.061	4.9%	0.0	9.9

[0050] At the higher add-on of 2.0 kg citral / ADMT fluff pulp shown in TABLE 1 as 5 and 6, it was not possible to keep the moisture increase under 1% for solution fractions of glycerol much under 85%. In other embodiments, a 100% water carrier has been used, such as in 3 and 6 shown in TABLE 1. It is possible to make a 100% water solution with relatively high concentrations of β -cyclodextrin at pH 12.5. Upon the addition of 1.5 moles of citral per mole of β -cyclodextrin, a fine, sprayable dispersion can be formed, in this case when the concentration of

complex is at or below about 19.7% At higher concentrations of complex, a coarse dispersion forms. This coarse dispersion settles in minutes and is very difficult to spray onto a substrate.

[0051] The cyclodextrin can be any cyclodextrin capable of forming cyclodextrin complexes with an odor-controlling compound. In certain embodiments, the cyclodextrin is a cyclodextrin containing six to twelve glucose units. For instance, cyclodextrins containing six to twelve glucose units can form inclusion complexes with many essential oils and odor control ingredients, wherein the essential oils and odor control ingredients can be released by the action of water and urine. Examples of acceptable cyclodextrins include, but are not limited to, commercially available, unsubstituted cyclodextrins (i.e. α -, β -, and γ -cyclodextrin). α -cyclodextrin contains 6 glucose units. β -cyclodextrin contains 7 glucose units. γ -cyclodextrin contains 8 glucose units. In particular, β -cyclodextrin can be used because β -cyclodextrin has a cost-performance ratio acceptable for use in most absorbent products.

[0052] As discussed above, the odor-controlling solution can form a cyclodextrin complex in solution with an odor-controlling compound. Unsubstituted cyclodextrins can be used to prepare such complexes. In general, three types of unsubstituted cyclodextrins are commercially available (i.e. α -, β -, and γ -cyclodextrin). Of these commercially available unsubstituted cyclodextrins, β -cyclodextrin has a cost-performance ratio acceptable for use in most absorbent products. The solubility of β -cyclodextrin in water at about 20° C. is only 1.8 g /100 ml water. However, the solubility of β -cyclodextrin can be increased to 75 g /ml at 20° C. at a pH of 12.5 (pK_a =12.2). The solubility of β -cyclodextrin in glycerol is 4.3 g /100 ml at 20° C. As shown in TABLE 2, the solubility of β -cyclodextrin and complexes of β -cyclodextrin/citral at elevated pH and temperature in an 85%/15% glycerol/water solution is of the order of 21.6 g /100 g solution or more. Solutions can also be made with propylene glycol. The solubility of β -cyclodextrin is greater in propylene glycol than in glycerol. Therefore, the solubility of cyclodextrin and complexes thereof can be increased by using a polar organic solvent/water solution, increasing the temperature of the solution, and increasing the pH.

[0053] [TABLE 2]

Solubility (g/100 g water)				
Temp	α	β		Υ
		pH 6.5	pH 12.5	
25	12.8	1.8	75	25.6
60	66.2	9.1	--	129.2

[0054] Furthermore, even though substituted cyclodextrins have greater water solubility than unsubstituted cyclodextrins, the performance of unsubstituted cyclodextrins remains very good.

- 5 The unsubstituted cyclodextrins can also be formulated into solutions at relatively high concentrations. Therefore, unsubstituted cyclodextrins can be used in the present solution.

[0055] The odor-controlling compounds can be any odor-controlling compound capable of providing odor control and/or odor masking. Examples of acceptable odor-controlling compounds include, but are not limited to, essential oils, such as citral, limonene, lavandin, geranium, persimmon, lemon, grapefruit, linalool, citronellol, linalyl acetate, geraniol, eugenol, floracetate, cardamom, eucalyptus, juniper berry, lavender, lemon verbena, lemongrass, patchouli, rose, spearmint, tea tree, oregano and mixtures thereof, botanical extracts such as Actiphyte™ by Lubrizol, odor-controlling substances, such as benzaldehyde, cinnamic aldehyde, ligustral, florhydral and mixtures thereof, and antimicrobial substances, such as eucalyptus, lavender, thyme, peppermint, cajuput, cinnamin, clove, sage, tea tree oils, and mixtures thereof. In particular, citral (i.e., C₁₀H₁₆O) can be used as an odor-controlling compound. Citral is an essential oil having a strong lemongrass odor that is effective to control and/or mask the odor of urine. Other scented essential oils, particularly limonene and persimmon, reactive aldehydes, particularly benzaldehyde, and thio-reducing compounds, particularly florhydral and ligustral, and fatty alcohols such as octanol, can be effective alone or in combination.

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[0056] The combined amount of the cyclodextrin and the odor-controlling compounds in the solution is limited by the solubility of the formed complex and is typically in the range of about 22 wt% or less for glycerol/water solutions in a ratio of about 85/15. In certain embodiments, the amount of the β -cyclodextrin/citral complex in the solution can be 15 wt% to 22 wt%, 20

wt% to 22 wt%, and/or about 21 wt%. In other embodiments, i.e., for a 100% water solution when a fine precipitate forms upon addition of citral to a β -cyclodextrin/water solution, the concentration for the complex can be lower, such as in the range of about 18 wt% or less. At higher concentrations, an undesirably coarse precipitate forms. The solution concentration of the complex is also important in reducing solution add-on so as to keep the moisture increase of the substrate below about 1 wt%. However, the ratio of odor-controlling compound to cyclodextrin is more important for imparting good odor control performance to a substrate. For citral, the optimum molar ratio of citral to β -cyclodextrin is about 1.5-1.75 moles of citral to 1.0 mole of β -cyclodextrin. At molar ratios of about 1:1 and less, odor control performance is much reduced (at equal add-ons of citral). At molar ratios of 1.75:1 and greater, volatile, uncomplexed citral imparts unnecessary color and scent to the substrate. In general, the molar ratio of odor-controlling compounds to cyclodextrin be can be, for example, 2.5:1, 2.25:1, 2.0:1, 1.75:1, 1.5:1, 1.4, 1.3, 1.2, 1.1, and 1.0.

[0057] A higher mole ratio of citral to β -cyclodextrin allows for less use of glycerol and β -cyclodextrin to achieve a target add-on of citral (kg citral per air-dried metric ton of fluff). It also reduces the moisture increase of the fluff pulp, as shown in TABLE 3 below. The molecular weights of β -cyclodextrin and citral are 1135 g/mole (dry) and 152 g /mole, respectively. The use of ActiphytesTM, Lubrizol Inc., and other more concentrated botanical essences could enable the loading of β -cyclodextrin with even higher levels of odor control compounds.

[0058] [TABLE 3]

Target Citral Add-On (kg citral /ADMT fluff)	Mole Ratio (citral / β -cyclodextrin)	Moisture Increase	Glycerol Add-On (kg /ADMT fluff)	β -cyclodextrin Add-On (kg /ADMT fluff)
0.4	1	0.30%	15.1	3.1
0.4	1.5	0.10%	7.4	2

[0059] In addition, commercially available complexes, such as a citral / β -cyclodextrin complex (Wacker Chemical, CAVAMAX® W7 CITRAL Complex), can be used to impart good odor control performance to fluff pulp. The commercially available complexes are believed to have a minimum 8.5 wt% citral content, which suggests a minimum citral: β -cyclodextrin mole ratio of

0.7:1. The target value of the citral:β-cyclodextrin mole ratio is unknown but it is likely to be in the range of 1.0:1. The odor-controlling solution can have a pH of greater than or equal to 12.5, 12, 11, 10, or 9.6. Below a pH of about 12, odor-control performance is greatly reduced. In certain embodiments, the pH is about 12.5.

5 [0060] The odor-controlling solutions described herein allow for a cyclodextrin complex to be precipitately deposited from the odor-controlling solutions and rapidly release odor-masking scents when hydrated with urine. Typical odor-controlling compounds are scented essential oils that are released from the cyclodextrin complex upon hydration of the treated fiber or film surface. The odor-controlling solution of the present disclosure can impart odor control to
10 materials used in absorbent products. The odor-controlling solution uses rapid-acting odor-masking and odor-control agents, e.g., odor-controlling compounds, that can be incorporated into a cyclodextrin complex and deposited on fluff fibers. These fluff fibers, also known as cellulose fluff pulp, can be used as an absorbent filler material for absorbent, such as diapers, bed pads, etc. The active ingredients (i.e., the odor-controlling compounds) can be stabilized in the
15 cyclodextrin complex for long shelf life and released only when the fibers contact urine. Essential oil fragrances and organic odor-control agents can be used to make stable cyclodextrin complexes that are highly soluble in polar organic and water solvent systems. These soluble complexes can be uniformly sprayed or otherwise applied to sheets of fluff fibers. *In situ* precipitation of the cyclodextrin complexes on the fiber surface after cooling to room
20 temperature provides unexpectedly efficient use of the odor-masking agents and delivers excellent odor control when the fibers become hydrated with urine. The odor-controlling solution of the present disclosure has: (1) a sufficient concentration of a cyclodextrin complex to impart urine odor control when applied to a material used in an absorbent product; and (2) an added amount of water that is low enough so as not to require an additional drying step after its
25 application.

[0061] The odor-controlling solution of the present disclosure for imparting odor control to materials used in absorbent products can contain a polar organic solvent, water, cyclodextrin, and one or more odor-controlling compounds. Cyclodextrin complexes containing a cyclodextrin and odor-controlling compounds have low water solubility and are difficult to uniformly apply

and be retained on the surface of a solid fiber or film. Solutions comprised of a polar organic solvent and water at elevated pH and temperature provide the capability of forming a cyclodextrin complex in solution. Such odor-controlling solutions also provide the solubility and solution stability required for uniform application of a homogeneous solution of a cyclodextrin complex onto a solid surface.

[0062] Substrate Having An Odor Controlling Solution Applied Thereupon

[0063] The substrate of the present disclosure comprises an odor-controlling solution applied thereupon. The odor-controlling solution is described in detail above, the discussions of which are incorporated herein by reference.

[0064] The substrate comprises at least 0.2 kg of the odor-controlling compound per 1000 kg of the substrate. In certain embodiments, the substrate comprises at least 0.3 kg of the odor-controlling compound per 1000 kg of the substrate. In other embodiments, the substrate comprises at least 0.6 kg, 0.8 kg, 1 kg, 1.5 kg, or 2 kg of the odor-controlling compound per 1000 kg of the substrate.

[0065] Also, the substrate can have an increase in moisture content of less than 1 wt. % as a result of the odor-controlling solution having been applied thereupon. That is, even after applying a sufficient amount of the odor-controlling solution to achieve at least 0.2 kg of the odor-controlling compound per 1000 kg of the substrate, the substrate still can have an increase in moisture content of less than 1 wt. %, as measured by comparing the weight of the substrate before application of the odor controlling solution and after the application of the odor controlling solution.

[0066] The odor-controlling solution discussed above provides cyclodextrin complexes containing a cyclodextrin and odor-controlling compounds. By utilizing a polar organic solvent and water at elevated pH and temperature, the cyclodextrin complex can be provided in a homogeneous solution and applied to the substrate. The cyclodextrin complexes precipitate *in situ* on the substrate after cooling to room temperature, but the substrate does not have a substantial increase in moisture content. In certain embodiments, the substrate can have an increase in moisture content of less than 0.8 wt. % as a result of the odor controlling solution

having been applied thereupon. In certain embodiments, the substrate can have an increase in moisture content of less than 5 wt. %, 4 wt. %, 3 wt. %, 2 wt. %, 0.6 wt. %, 0.4 wt. %, 0.2 wt. %, 0.13 wt. %, or 0.1 wt. %. There is a significant practical and economic value to limiting the moisture content of the substrate to less than about 1%. The addition of a drying step to a typical fluff pulp machine would require capital in the range of \$30-40 million. Therefore, it is desirable to reduce the moisture content to the extent possible.

[0067] The substrate is not limited to any particular substrate material. For instance, the substrate can be a fluff pulp in the form of a compressed sheet or an air-laid fiber form, a synthetic or cellulosic fiber spun-bond or carded nonwoven, woven fabric, solid material, or an absorbent article. When the substrate is the solid material, the solid material is selected from the group consisting of a polymeric foam, leather, and plastic film. When the substrate is an absorbent article, the absorbent article is an adult incontinence control pad, feminine hygiene pad, pull-up underwear, adult brief, baby diaper, or bed pad.

[0068] Article Comprising The Substrate Having An Odor Controlling Solution Applied Thereupon

[0069] The articles of the present disclosure comprise the substrate discussed above, the discussion of which is incorporated herein by reference. The article is not limited, but can include, for example, articles that would benefit from the improved odor control achieved by the present disclosure. For instance, such articles can include absorbent articles such as an adult incontinence control pad, feminine hygiene pad, pull-up underwear, adult brief, baby diaper, or bed pad. Other substrates include, but are not limited to, fluff pulp in compressed sheet or air-laid fiber form, synthetic or cellulosic fiber spunbond or carded nonwoven, woven fabric, or solid material such as polymeric foam, leather, or plastic film.

[0070] Method Of Preparing An Odor Controlling Substrate

[0071] The odor-controlling substrate can be prepared by providing a substrate, applying an odor-controlling solution onto the substrate to obtain the odor-controlling substrate. The substrate and the odor-controlling solution are described in detail above, the discussions of which are incorporated herein by reference.

[0072] In certain embodiments, the odor-controlling solution is applied onto the substrate at elevated temperature and pH. By “elevated temperature,” the disclosure means that the temperature is greater than room temperature. For example, the odor-controlling solution can be applied onto the substrate at an elevated temperature of 70 °C to 90 °C. In certain embodiments, the odor-controlling solution can be applied onto the substrate at an elevated temperature 75 °C to 90 °C, 80 °C to 90 °C, or 83 °C to 86 °C.

[0073] In an exemplary embodiment, a fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials. A warm solution containing citral was sprayed onto a sheet of fluff pulp at a solution add-on of about 0.055 g of odor-controlling solution per g of fluff. The sheet was not dried after the application of the odor-controlling solution. The citral add-on was calculated to be 2.0 kg citral per air-dried metric ton of fluff. The moisture content of the fluff pulp increased by 0.65%, i.e., increased from 8% moisture to 8.65% moisture. It can be beneficial to keep the increase in moisture content to a level less than 5% or even less than 1%.

[0074] Complexation of citral with β -cyclodextrin reduces evaporation of the citral when dry, but allows release of a citral scent when the complex is hydrated with water. The exemplary embodiment had no detectable scent when dry but had an immediate and pronounced release of lemon scent when hydrated with only 2.5 g of water per g of fluff. Furthermore, this exemplary embodiment effectively masked the scent of warm urine (at 35°- 40° C) using 2.5 g of urine per g of fluff to 10 g of urine per g of fluff.

[0075] Other Advantages Imparted to Fluff Pulp by the Solution

[0076] Glycerol used in the formulation of the solution can impart additional benefits to fluff pulp such as reduced sheet Mullen and ease of compressibility of fiberized fluff pulp.

[0077] For instance, a sheet of RAYFLOC® pulp sprayed on one side with 0.08 g solution/g pulp of a solution of 85 wt.% glycerol and 15 wt.% water at 80°C. reduced Mullen from an average value of 1472 kPa to 1107 kPa. Bursting strength (or Mullen) is the hydrostatic pressure, applied at a controlled rate, required to produce a rupture of a conditioned test specimen. To obtain the measurement of bursting strength (or Mullen), pressure is applied to a

circular area of the test specimen, 30.48 mm (1.20 inches) in diameter, at a controlled rate through a rubber diaphragm. The material being tested is initially flat and held rigidly at the circumference of the test location. The specimen is free to bulge and eventually burst as the pressure is increased. Bursting strength (or Mullen) is recorded as the pressure in kPa at rupture.

- 5 [0078] A similar treatment was found to improve the mechanical properties of an air-laid sheet of fluff pulp. The mechanical properties related to softness and handle are KES shear modulus and compression energy. The shear modulus of fluff pulp at a basis weight of 400 g/sm and a density of 0.37 g/cc was reduced by 20%, and the compression energy was reduced by 29%. These values indicate that an air-laid core of OCF (Odor Control Fiber) might be perceived as
- 10 being softer than untreated fluff pulp. The Kawabata Evaluation System (KES) is used to make objective measurements of hand properties. With low forces applied, the KES instrument measures mechanical properties that correspond to the fundamental deformation of fabrics in hand manipulation. Samples are conditioned and tests performed at laboratory atmosphere conditions of $21^{\circ} \pm 2^{\circ}$ C. and $65\% \pm 5\%$ RH. Samples had a width of 20 cm. Three replications
- 15 are obtained for shear stiffness. In shear testing, the KES-FB1 Tensile-Shear Tester applies opposing, parallel forces to the specimen, until a maximum offset angle of 0.5° is reached. A pretension load of 50 gf/cm was applied. A lower value of shear stiffness (gf/cm*degree) indicates less resistance to shearing movement corresponding to a softer material.

[0079] Examples

- 20 [0080] The features and advantages of the solutions containing cyclodextrins for imparting odor control to materials used in absorbent products and associated products and methods are more fully shown with respect to the following illustrative examples and embodiments.

[0081] Example 1: Preparation of Cyclodextrin Complex in Solution

- [0082] In the following example, β -cyclodextrin was obtained from Acros Organics (CAS:7585-39-9) and Wacker Chemical under the trade name W7. Citral is a volatile lemongrass-scented essential oil that was obtained from Perfumer's Apprentice (CAS:5392-40-5).
- 25

[0083] A solution was prepared by first blending 85 wt.% glycerol and 15 wt.% water and increasing its pH to 12.5 with sodium hydroxide. 50 g. of this mixture was heated after the pH adjustment. At about 75° C., 11.5 g. of β -cyclodextrin was added. Heating and stirring continued until the β -cyclodextrin was completely dissolved at around 86° C. At that point, 2.3 g. of citral was added (using a mole ratio of 1.5 moles citral/mole β -cyclodextrin) with continued stirring for a few minutes until a clear, water-white solution was obtained. This solution contained 21.6 g of β -cyclodextrin and citral per 100 g of solution, 66.7 g of glycerol per 100 g solution, and 11.8 g of water per 100 g. solution. The water content of the β -cyclodextrin (up to 10% by weight) was not considered in these calculations.

10 [0084] Example 2: pH Determination

[0085] In the following example, the pH of untreated RAYFLOC® fluff from Rayonier Performance Fibers, LLC was compared to an exemplary embodiment, 31-5 OCF (Odor Control Fiber), that was treated with a solution of the present disclosure. The solution applied to the 31-5 OCF sample contained 66.6 wt.% glycerol, 11.8 wt.% water, 18.0 wt.% β -cyclodextrin, and 3.6 wt. % citral (i.e., 1.5:1 citral:cyclodextrin molar ratio). The pH of the glycerol/water solution was adjusted to a pH of 12.5 at room temperature using sodium hydroxide. The 31-5 OCF sample was made using a solution add-on of 0.011 grams solution per grams of fluff at 86° C. to provide a citral add-on of 0.4 kg citral per air-dried metric ton of fluff. Using the test method below, the 31-5 OCF sample had a pH of 5.8 and the untreated RAYFLOC® fluff had a pH of 6.0, as shown in TABLE 4 below. The use of a relatively high pH solution had a negligible effect on the pH of the 31-5 OCF.

[0086] [TABLE 4]

	pH
Deionized water	6.5 – 7.0
RAYFLOC® fluff pulp	6
31-5 OCF	5.8

[0087] The method for determining the pH of the fibers is provided as follows. The pH of the fiber was determined by placing two grams of a defiberized sample into a 500 mL Erlenmeyer flask. 100 grams of distilled water at pH 6.5-7.0 was added. The flask was glass-stoppered and shaken until a thick slurry was obtained. An additional 100 grams of distilled water was added to the flask before it was stopped and briefly shaken at about 15-minute intervals until the sample was completely slurried. After about one hour, about 50-mL of the slurry was poured into a clean 150 mL beaker. This portion was used to rinse the beaker and was discarded. The remainder of the slurry was then poured into a beaker and pH was determined with a previously calibrated pH meter.

[0088] Example 3: Dose/Response of Citral for Urine Odor Control

[0089] In the following example, the dose and response of citral for urine odor control in exemplary embodiments were determined.

[0090] Using the solution described in Example 2, exemplary embodiments were prepared whereby the amounts of citral add-on were varied over a range of 0.1 to 3.3 kg citral per air-dried metric ton of fluff. The citral add-on was varied by adjusting the solution add-on. Fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials of Jesup, GA. The following TABLE 5 summarizes the variations used to determine the dose and response of citral for urine odor control.

[0091] [TABLE 5]

	Citral:CD mole ratio	Citral add-on (kg/ADMT)	pH	Mix Temp (C.)	Appl'n Temp (C.)	TOTAL ODOR SCORE	Water (control) vs. Urine ODOR SCORE	Urine (control) vs. Urine ODOR SCORE
25-1	1.5	3.3	12.5	86°	86°	+48	+12	+36
31-1	1.5	2.0	12.5	86°	86°	+56	+24	+32
31-4	1.5	0.8	12.5	86°	86°	+52	+20	+32
31-5	1.5	0.4	12.5	86°	86°	+52	+24	+28
37-1	1.5	0.2	12.5	86°	86°	+38	+20	+18
37-2	1.5	0.1	12.5	86°	86°	-18	-36	+18
theoretical		0				-36	-36	0

[0092] As shown in FIG. 1, odor control performance measured as the TOTAL ODOR SCORE increased with increasing citral add-on up to a value of about 0.3 to 0.4 kg citral per air-dried metric ton of fluff and then remained constant up to a value of 3.3 kg citral per air-dried metric ton of fluff.

[0093] The resulting embodiments shown in TABLE 5 were evaluated according to the following method.

[0094] URINE ODOR ASSESSMENT OF OCF USING A BLIND, TRIANGULAR FORCED-CHOICE TEST METHOD WITH EXPERT PANELIST

[0095] SAMPLE PREPARATION -- Individual samples 60 mm in diameter were cut with a die from a sheet of air-laid fluff pulp that had a basis weight of $400 \text{ g/cm}^2 \pm \text{about } 5 \text{ g/cm}^2$ and a density of about 0.06 g/cc . For each odor test, eight samples of a control material and six samples of an unknown, or test material, were made. The samples were placed in cylindrical carbonate or glass jars 70 mm in diameter and 70 mm in height. The volume of a jar was about 270 cm^3 . Each jar had a plastic screw-on lid. One sample was placed in each jar and dosed with either distilled water or fresh urine and heated in a water bath at $40^\circ \text{ C.} \pm 5^\circ \text{ C.}$ during the test. Dosing was done with a calibrated syringe taking care to evenly distribute the liquid over the sample. Between tests, the jars were washed with an unscented dishwashing liquid.

[0096] TEST DESIGN -- There were two parts to an odor test. In Part 1, one test sample was dosed with urine and compared to two control samples dosed with distilled water. In Part 2,

another test sample that had been dosed with urine was compared to two control samples also dosed with urine. TABLE 6 below provides a comparison.

[0097] [TABLE 6]

		time (min)	Dilution Level (g liq / g fluff)		
			2.5	5	10
PART 1					
2 Control samples with water vs. 1 test sample with urine		10	#1	#2	#3
		100	#7	#8	#9
PART 2					
2 Control samples with urine vs. 1 test sample with urine		10	#4	#5	#6
		100	#10	#11	#12
			<i>Tests #1 - #12, in chronological order, for assessment of each test sample.</i>		

- 5 [0098] For each test sample, there are twelve odor comparisons. There are two parts to the test and three levels of urine dosing (i.e. 2.5, 5, and 10 g of liquid per g. of dry sample) in each part at both 10 and 100 min after dosing. Samples were maintained at a temperature of 40° C. ± 5° C. during the test. In Part 1 of the test above, for convenience, the same two control samples with 10 g. water / g. fluff were compared to three separate test samples prepared at the three dilution
- 10 levels of urine. Only two control samples with water are used because the odor of these controls does not change after opening the jar or over time. After the odor test at 10 min., samples are returned to the water bath for an additional 100 min. before the final odor assessments. For Part 2 of the test, two control samples with urine were compared to one test sample with urine and separate samples were used for each comparison. In summary, six samples of the test material
- 15 were prepared with urine (for Parts 1 and 2 at each of the three dilution levels). Six of the control samples were prepared with urine (for Parts 1 and 2 at each of the three dilution levels) and an additional two control samples were prepared with distilled water (at 10 g water /g fluff) for use in Part 1.

- 20 [0099] PANELISTS -- Each odor assessment was made by having a test administrator remove appropriate jars from the water bath and present them to the panelist blind. The panelist

removed the lid of each jar and sniffed the headspace of each jar before recapping. The panelist was instructed to select the one of the three jars that was different from the other two, even if they had to guess. This statistical approach is called “triangular forced-choice”. The panelist would tell the test administrator which sample was different, and whether their selection was a “detection” (i.e., one is confident that their selection was different from the other two) or was a “guess” (i.e., one is not confident that their selection was different from the other two). If the panelist makes a correct selection, and that selection is a “detection”, the test administrator records a value of “6” for that comparison. If the panelist guessed at the selection, a value of “2” was recorded for a correct guess and a value of “0” was recorded for an incorrect guess. TABLE 7 below provides the response key.

[0100] [TABLE 7]

Response Key			
		0	Incorrect guess
+ unknown better than control		2	Correct guess
- unknown worse than control		5	Incorrect detection
		6	Correct detection

[0101] Odor values of 2 and 6 are used because sensory responses, such as odor, follow a power law, where the apparent odor intensity increases as a power function of the dosing or odor concentration, i.e., according to the following:

$$I = k C^n \quad (1)$$

and

$$\log_{10} I = n \log_{10} C + \log_{10} K \quad (2)$$

where I is the odor intensity or strength, C is a measure of the mass concentration of odorant, and k and n are constants that are different for every odorant. Equation (2) is linear plotted on a log-log scale. The dilution levels in the test, which double on each step, are related to C, the mass concentration of odorant. Using a $\log_{10}$ transformation forces an equal spread (i.e. 0.301) between the levels of 2.5, 5, and 10 g. liquid /g. fluff. An equal spread is forced between upper

and lower values of I, or the Odor Score, in the test. So, $\log_{10}(2 * 0.301) = 4$, i.e., the spread between Odor Score values of 6 and 2.

[0102] The panelist also provides the test administrator with an assessment of the pleasantness or unpleasantness of the odor of the test sample relative to that of the odor of the control samples.

5 For example, if the panelist makes a correct selection, and is certain of that selection (i.e. it was a “detection”), and the odor of the selected test sample was more pleasant than that of the control samples, the test administrator would record a value of “+6” for that comparison. The test administrator would record a value of “-6” if that test sample was more unpleasant than that of the control samples.

10 [0103] There is one other situation that can occur in the test. If the scented test sample with urine can be confidently identified relative to the control samples with water, but the test sample with urine has no detectable urine odor, the result is recorded as “±6” indicating that either odor is subjectively acceptable. For example, a lemongrass scent with no detectable urine odor can be as acceptable as a sample with no scent at all. The choice depends on the preference of the
15 panelist. In adding up the odor scores, a “±6” is scored as a “+6” because it is as good as an untreated control with distilled water.

[0104] Panelists for an odor test can be recruited from the community at large. A person who uses tobacco, is pregnant, or has chronic allergies or asthma or nasal congestion is not a candidate for the odor panel. Panelists must be free of colds and other respiratory ailments for
20 the test and must not chew gum or eat 30 minutes before a test. Panelists must not wear perfume, cologne, aftershave, or scented deodorant on the day of a test. Samples are prepared for each panelist using fresh urine collected from that panelist on the morning of the day of the odor test. Panelists are only required to smell their own urine. Urine is discarded at the end of each day.

25 [0105] ANALYSIS OF ODOR SCORES -- Examples below illustrate the calculation and analysis of the Odor Scores. The first example is a test of OCF 31-4 vs. untreated RAYFLOC® fluff pulp. That is, RAYFLOC® fluff pulp was the control sample in this odor test.

[0106] [TABLE 8]

								TOTAL ODOR SCORE
Control (untreated fluff) Unknown (31-4 OCF sample)		time (min)	Dilution Level (g liq / g fluff)			Subtotal		
			2.5	5	10			
PART 1								
2 untreated fluff samples (control) with water vs. 1 test sample of 31-4 with urine		10	±6	±6	±2	+14	Water (control) vs. Urine (unknown) ODOR SCORE +20	+52
		100	±6	±6	-6	+6		
PART 2								
2 untreated fluff samples (control) with urine vs. 1 test sample of 31-4 with urine		10	+6	+6	+6	+18	Urine (control) vs. Urine (unknown) ODOR SCORE +32	
		100	+6	+6	+2	+14		

[0107] In Part 1 of the test, at 2.5 g. and 5 g. urine / g. 31-4 OCF sample (10 min.), the Odor Scores were ±6. This indicated that there was no detectable urine odor on the 31-4 sample vs. untreated fluff with distilled water. At 10 g. urine / g. OCF sample, a lemongrass scent was barely detectable but there was still no detectable urine odor. At 10 g. urine / g. 31-4 OCF sample and 100 min. there was detectible urine odor on the 31-4 sample, and an Odor Score of “-6” was recorded.

[0108] In Part 2 of the test, the odor of the 31-4 OCF sample with urine was detectable (and more pleasant) relative to untreated RAYFLOC® with urine (i.e. “+6” values). At 100 min. and the highest level of urine dosing (i.e. 10 g. urine / g. fiber), the panelist had to guess but guessed correctly that the 31-4 OCF sample was different, and that its odor was more pleasant.

[0109] The second example is a test of the 31-4 OCF sample vs. itself. That is, the 31-4 OCF sample was the control sample in this odor test.

[0110] [TABLE 9]

								TOTAL ODOR SCORE
Control (31-4 OCF sample) Unknown (31-4 OCF sample)		time (min)	Dilution Level (g liq / g fluff)			Subtotal		
			2.5	5	10			
PART 1								
2 test samples of 31-4 with water vs. 1 test sample of 31-4 with urine		10	-2	-6	-6	-14	Water (control) vs. Urine (unknown) ODOR SCORE -32	-32
		100	-6	-6	-6	-18		
PART 2								
2 test samples of 31-4 with urine vs. 1 test sample of 31-4 with urine		10	0	0	0	0	Urine (control) vs. Urine (unknown) ODOR SCORE 0	
		100	0	0	0	0		
			Tests #1 - #12, in chronological order, for assessment of each test sample					

[0111] Odor Score values were negative in the first part of the test. This may be a result of it being easier to detect a slight urine odor for a scented 31-4 sample with urine vs. a scented 31-4 sample with water than it was to detect a urine odor in the first example for a scented 31-4 sample with urine vs. an unscented fluff sample with water. All values in the second part of the test were “0”. All of the samples were the same and the panelist had to guess which one was arbitrarily designated as the test sample. The panelist guessed wrong in all cases, although there was a 33% chance of randomly making a correct choice.

[0112] The Odor Score obtained from Part 1 of the test is a comparison of an unknown material with urine vs. a control material with distilled water. The Odor Score obtained from Part 2 of the test is a comparison of the same unknown material with urine vs. the same control material with urine. The TOTAL ODOR SCORE is the sum of the odor scores from Parts 1 and 2 of the test. Variations of the test can be used with samples containing superabsorbent polymer and samples cut from absorbent cores and absorbent products, as well as using other reference materials for controls. The Odor Scores provide a good semi-quantitative measure of the effectiveness of odor control of the invention.

[0113] Example 4: Odor Control Performance of a Citral/ β -cyclodextrin Complex

[0114] The following example characterizes the unexpected discovery that the odor control performance of a β -cyclodextrin/citral complex can be improved by forming the complex in solution at pH 12.5 and applying that solution at elevated temperature such that the complex precipitates *in situ* on the fiber surface at room temperature. The following example also characterizes the unexpected discovery that the odor control performance of the citral: β -cyclodextrin complex exhibited an optimum at a mole ratio of citral: β -cyclodextrin of about 1.5:1.

[0115] The following TABLE 10 shows the performance of exemplary citral/ β -cyclodextrin embodiments with decreasing add-on of citral (kg citral per air-dried metric ton fluff) using citral: β -cyclodextrin mole ratios of 1.0:1, 1.5:1, and 1.75:1. Additionally, control samples used to generate odor scores were made of untreated RAYFLOC® fluff from Rayonier Performance Fibers, LLC. The odor score was determined by the triangular forced-choice test method discussed above. As seen in the following TABLE 10, the odor control performance was best for a citral: β -cyclodextrin mole ratio of 1.5:1, and was independent of citral add-on over a range of 3.3 to 0.4 kg citral /ADMT fluff pulp at that mole ratio. A citral: β -cyclodextrin mole ratio of 1.75:1 had a slightly detectable scent and color on fluff pulp, indicating that not all of the citral was complexed with the cyclodextrin.

[0116] [TABLE 10]

ODOR SCORE (vs. Rayfloc) AS A FUNCTION OF CITRAL : β -CYCLODEXTRIN MOLE RATIO											
Sample No.	Sample ID	Citral : β -CD mole ratio	Solution pH	Mix Temp (C.)	Appl'n Temp (C.)	Appearance of Solution	Add-On (kg citral /ADMT fluff)	TOTAL ODOR SCORE	Water (control) vs. Urine (unknown) ODOR SCORE	Urine (control) vs. Urine (unknown) ODOR SCORE	Control in Odor Test
1	78-2	1.0 : 1	9.6	83°	83°	CLEAR	3.2	-16	-36	+20	vs. Rayfloc
2	78-1	1.0 : 1	9.6	83°	83°	CLEAR	2.2	-28	-36	+8	vs. Rayfloc
3	68-1	1.0 : 1	12.5	85°	85°	CLEAR	2.0	+16	-12	+28	vs. Rayfloc
4	53-1	1.0 : 1	12.5	87°	87°	CLEAR	1.4	-26	-36	+10	vs. Rayfloc
5	25-1	1.5 : 1	12.5	86°	86°	CLEAR	3.3	+48	+12	+36	vs. Rayfloc
6	31-1	1.5 : 1	12.5	86°	86°	CLEAR	2.0	+56	+24	+32	vs. Rayfloc
7	31-4	1.5 : 1	12.5	86°	86°	CLEAR	0.8	+52	+20	+32	vs. Rayfloc
8	31-5	1.5 : 1	12.5	86°	86°	CLEAR	0.4	+52	+24	+28	vs. Rayfloc
9	37-1	1.5 : 1	12.5	86°	86°	CLEAR	0.2	+38	+20	+18	vs. Rayfloc
10	37-2	1.5 : 1	12.5	86°	86°	CLEAR	0.1	-18	-36	+18	vs. Rayfloc
11	72-1	1.75 : 1	12.5	85°	85°	CLEAR	0.8	+60	+24	+36	vs. Rayfloc
12	60-1	1.75 : 1	12.5	86°	86°	CLEAR	0.4	-14	-24	+10	vs. Rayfloc

[0117] The best odor control performance was achieved by using a citral: β -cyclodextrin mole ratio of 1.5:1 and a solution pH of 12.5. TABLE 11 below shows the performance of exemplary citral/ β -cyclodextrin embodiments made with a citral: β -cyclodextrin mole ratio of 1.5:1 as a function of solution pH, mix temperature, and application temperature. The first group of three samples was made with a solution that was mixed and applied at an elevated temperature of 86° C. For the second group, the solution was mixed at elevated temperature but allowed to cool and applied at room temperature. The solution was mixed and applied at room temperature for the third group. Odor scores increased (i.e., performance improved) with increasing pH for each group. There was no significant difference between groups as a result of normal variation in the odor test scores.

[0118] [TABLE 11]

ODOR SCORES (vs. 31-4) AS A FUNCTION OF pH, MIX TEMP AND APPLICATION TEMP AT 1.5 : 1 Citral : CD MOLE RATIO											
Sample No.	Sample ID	Citral : β -CD mole ratio	Solution pH	Mix Temp (C.)	Appl'n Temp (C.)	Appearance of Solution at Appl'n Temp	Active Add-On (kg citral /ADMT fluff)	TOTAL ODOR SCORE	Water (control) vs. Urine (unknown) ODOR SCORE	Urine (control) vs. Urine (unknown) ODOR SCORE	Control in Odor Test
1	31-4	1.5 : 1	12.5	86°	86°	CLEAR	0.8	-32	-32	0	vs. 31-4
2	58-1	1.5 : 1	9.6	86°	86°	CLEAR	0.8	-46	-36	-10	vs. 31-4
3	55-1	1.5 : 1	6.5	86°	86°	CLEAR	0.8	-54	-36	-18	vs. 31-4
4	No sample made	1.5 : 1	12.5	86°	20°	ORANGE-BROWN ON COOLING	0.8	--	--	--	--
5	45-1	1.5 : 1	9.6	86°	20°	MILKY WHITE	0.8	-36	-32	-4	vs. 31-4
6	43-1	1.5 : 1	6.5	86°	20°	MILKY WHITE	0.8	-54	-36	-18	vs. 31-4
7	51-3	1.5 : 1	12.5	20°	20°	MILKY WHITE	0.8	-22	-30	+8	vs. 31-4
8	62-1	1.5 : 1	9.6	20°	20°	MILKY WHITE	0.8	-32	-36	+4	vs. 31-4
9	67-1	1.5 : 1	6.5	20°	20°	MILKY WHITE	0.8	-66	-36	-30	vs. 31-4

[0119] The control sample used to generate the odor scores in TABLE 11 was a 31-4 sample as used in Example 3. A 31-4 sample was also tested as Sample No. 1 in TABLE 11 above. All of the water (i.e., control) vs. urine odor scores were negative because it is easier to detect a slight difference in urine malodor between similarly scented samples than it is to detect a slight urine malodor in a scented sample vs. an unscented sample such as RAYFLOC® fluff treated with water. The 31-4 sample was favored because the complex formed a clear, homogeneous solution at an elevated temperature of about 86° C. This step did not improve the odor control performance of the complex. However, it greatly improved the ability to handle the solution and uniformly apply it to a substrate surface. A solution of a β -cyclodextrin/citral complex sprayed on fluff pulp at an elevated temperature of about 86° C. would precipitate *in situ* on the fiber surface as it cooled to room temperature.

[0120] Turbidity is an indicator of the solubility of the complex in solution. A soluble complex forms a clear, water-white solution. An insoluble complex forms an opaque, milky-white dispersion that can settle over time. A milky-white dispersion is formed if a solution at 86° C. cools to room temperature or if no heat is used in the mixing or application steps. A simulated laboratory kiss roll method was used to apply the milky-white dispersions of the complex to fluff pulp. A known amount of the milky-white dispersion was applied to a glass surface and a fluff pulp sheet was immediately placed in contact with the glass surface before the dispersion dried. Wet pick up of the dispersion was estimated by immediately weighing the sheet of fluff pulp.

[0121] Without being bound to a singular and complete understanding of the physical-chemical modifications imparted by the interaction of a citral/ β -cyclodextrin complex with solution pH, the inventors believe that the unique physical form of the precipitate formed on a solid cellulosic surface may lead to the unexpected performance enhancement in odor control for samples made using an elevated solution pH of 12.5. FIG. 2 and FIG. 3 are photomicrographs below showing the particle size of citral/ β -cyclodextrin complexes precipitated from 86° C. glycerol/water solutions at pH 12.5 and 9.6 after the solutions cooled to room temperature.

[0122] Solid particles of the complex precipitated from a 12.5 pH solution were uniform in dimension and about 50 μm in diameter. Particles precipitated from solution at pH 9.6 had dimensions in the range of 100 – 400 μm . It was an unexpected discovery to learn that the odor

control performance of a β -cyclodextrin/citral complex could be improved by forming the complex in a glycerol/water solution at a citral: β -cyclodextrin mole ratio of about 1.5:1 and at an elevated pH of about 12.5.

[0123] Example 5: Substrate Preparation

- 5 [0124] A sample was prepared using an 85/15 ratio of glycerol/water and a pH adjustment of 9.6 using β -cyclodextrin from Acros Organics and citral, a volatile lemongrass-scented essential oil, from Perfumer's Apprentice. The solution was mixed and applied at a temperature of 86° C. At the application temperature of 86° C, the components formed a clear, water-white solution. The citral to β -cyclodextrin mole ratio was 1.0. The amount of citral added on was 2.4 kg of citral
- 10 per air-dried metric ton ("ADMT") of fluff. Fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials Jesup, GA. The calculated increase in moisture content of the substrate was 1.0 wt%. This was checked using a METTLER TOLEDO® Moisture Analyzer where the treated sample had a moisture content of $6.29\% \pm 0.18\%$ and the untreated Rayfloc control had a
- 15 moisture content of $5.33\% \pm 0.39\%$. An experimental value of 0.96% was obtained for the moisture increase of the sample. Because the moisture increase was less than about 1 wt% or less, no drying step was required. The overall odor score was -28 as determined by the blind, triangular forced-choice test method with an expert panel discussed above. This sample did not provide good odor control performance even for this relatively high amount of citral add-on.
- 20 Citral add-on was 2.4 kg of citral per air-dried metric ton ("ADMT") of fluff. Odor control performance was poor because the citral: β -cyclodextrin mole ratio was less than 1.5:1 and the solution pH was less than pH 12. Specifications and odor score measurements for Example 5 are given in TABLE 12 and TABLE 13.

[0125] [TABLE 12]

SPECIFICATION												
Sample	Description	Solution					Solution Add-on (g soln /g fluff)	Solids Add-On (g solids /g fluff)	CD Add-On (kg CD /ADMT fluff)	Citral Add-On (kg Citral /ADMT fluff)	Glycerol Add-On (kg glycerol /ADMT fluff)	Fluff Moisture Increase (%)
		Component	Liquid Conc	Soln Wt. Fraction	T (°C)	pH						
Example	β-CD/Citral complex	glycerol	0.85	0.658	86	9.6	0.088	0.020	17.6	2.4	58.2	1.0%
		water	0.15	0.116								
		Subtotal		0.774								
		β-Cyclodextrin		0.199								
		Citral		0.027								
		Total		1.00								
		Mole Ratio (mol CD/mol Citral) =				1.0						
		Solution Preparation:										
	MW (g/mol)		g used	%								
	1135	CD	12.9	19.9%	22.6%		Total	14.6				
	152.2	Citral	1.73	2.7%								
		Soln	50.0	77.4%								
		Total	64.6	100%								

[0126] [TABLE 13]

ODOR EVALUATION										
Triangular Forced-Choice Odor Evaluation			time (min)	Liquid Dosing Level (g liq / g fluff)			Odor Score Summary			
Test	2 Controls	1 Unknown		2.5	5	10				
1	Rayfloc/water	OCF/urine	10	6-	6-	6-		-18	-36	-28
			100	6-	6-	6-		-18		
2	Rayfloc/urine	OCF/urine	10	2+	2+	0		+4	+8	
			100	2+	2+	0		+4		

5 [0127] Example 6: Substrate Preparation

[0128] A sample was prepared using an 85/15 ratio of glycerol/water and a pH adjustment of 12.5 using β -cyclodextrin from Acros Organics and citral from Perfumer's Apprentice. The solution was mixed and applied at a temperature of 86° C. At the application temperature of 86° C. the components formed a clear, water-white solution. The citral to β -cyclodextrin mole ratio was 1.0. The amount of citral added on was 2.4 kg of citral per air-dried metric ton ("ADMT") of fluff. Fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials Jesup, GA. The calculated increase in moisture content of the substrate was 1.0 wt%. Because the moisture increase was less than about 1 wt% or less, no drying step was required. The overall odor score

was +16 as determined by the blind, triangular forced-choice test method with an expert panel discussed above. This sample provided better odor control than the sample in Example 5 because of an increase in solution pH from 9.6 to 12.5. Specifications and odor score measurements for Example 6 are given in TABLE 14 and TABLE 15.

5 [0129] [TABLE 14]

SPECIFICATION												
Sample	Description	Solution					Solution Add-on (g soln /g fluff)	Solids Add-On (g solids /g fluff)	CD Add-On (kg CD /ADMT fluff)	Citral Add-On (kg Citral /ADMT fluff)	Glycerol Add-On (kg glycerol /ADMT fluff)	Fluff Moisture Increase (%)
		Component	Liquid Conc	Soln Wt. Fraction	T (°C)	pH						
Example	β -CD/Citral complex	glycerol	0.85	0.638	86	12.5	0.080	0.020	17.6	2.4	51.2	0.9%
		water	0.15	0.113								
		Subtotal		0.751								
		β -Cyclodextrin		0.220								
		Citral		0.029								
		Total		1.00								
		Mole Ratio (mol CD/mol Citral) =										
		1.0										
		Solution Preparation:										
	MW (g/mol)		g used	%								
	1135	CD	14.6	22.0%	24.9%		Total	16.6				
	152.2	Citral	1.96	2.9%								
		Soln	50.0	75.1%								
		Total	66.6	100%								

[0130] [TABLE 15]

ODOR EVALUATION										
Triangular Forced-Choice Odor Evaluation			time (min)	Liquid Dosing Level (g liq / g fluff)				Odor Score		
Test	2 Controls	1 Unknown		2.5	5	10		Summary		
1	Rayfloc/water	OCF/urine	10	6±	6-	6-		-6	-12	
			100	6±	6-	6-		-6		
									+16	
2	Rayfloc/urine	OCF/urine	10	6+	6+	6+		+18		+28
			100	6+	2+	2+		+10		

[0131] Example 7: Substrate Preparation

- 10 [0132] A sample was prepared using an 85/15 ratio of glycerol/water and a pH adjustment of 12.5 using β -cyclodextrin from Acros Organics and citral from Perfumer's Apprentice. The solution was mixed and applied at a temperature of 86° C. At the application temperature of 86° C, the components formed a clear, water-white solution. The citral to β -cyclodextrin mole ratio was 1.5. The amount of citral added on was 2.0 kg of citral per air-dried metric ton ("ADMT")

of fluff. Fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials Jesup, GA. The calculated increase in moisture content of the substrate was 0.6 wt%. Because the moisture increase was less than about 1 wt% or less, no drying step was required. The overall odor score was +56 as determined by the blind, triangular forced-choice test method with an expert panel discussed above. This sample provided very good odor control performance as a result of a citral to β -cyclodextrin mole ratio of 1.5:1 and a solution pH of 12.5. Specifications and odor score measurements for Example 7 are given in TABLE 16 and TABLE 17.

[0133] [TABLE 16]

SPECIFICATION												
Sample	Description	Solution					Solution Add-on (g soln)	Solids Add-On (g solids)	CD Add-On (kg CD)	Citral Add-On (kg Citral)	Glycerol Add-On (kg glycerol /ADMT fluff)	Fluff Moisture Increase
		Component	Liquid Conc	Soln Wt. Fraction	T (°C)	pH						
Example	β-CD/Citral complex	glycerol	0.85	0.666	86	12.5	0.055	0.012	9.9	2.0	36.8	0.6%
		water	0.15	0.118								
		Subtotal		0.784								
		β-Cyclodextrin		0.180								
		Citral		0.036								
	Total			1.00								
		Mole Ratio (mol CD/mol Citral) = 1.5										
		Solution Preparation:										
	MW (g/mol)		g used	%								
	1135	CD	11.5	18.0%	21.6%		Total	13.8				
	152.2	Citral	2.31	3.6%								
		Soln	50.0	78.4%								
		Total	63.8	100%								

10

[0134] [TABLE 17]

ODOR EVALUATION										
Triangular Forced-Choice Odor Evaluation			time (min)	Liquid Dosing Level (g liq / g fluff)			Odor Score Summary			
Test	2 Controls	1 Unknown		2.5	5	10				
1	Rayfloc/water	OCF/urine	10	6±	6±	6±		+18	+24	+56
			100	6±	6±	6-		+6		
2	Rayfloc/urine	OCF/urine	10	6+	6+	6+		+18	+32	
			100	6+	6+	2+		+14		

[0135] Example 8: Substrate Preparation

[0136] A sample was prepared using an 85/15 ratio of glycerol/water and a pH adjustment of 12.5 using β -cyclodextrin from Acros Organics and citral from Perfumer's Apprentice. The solution was mixed and applied at a temperature of 86° C. At the application temperature of 86° C. the components formed a clear, water-white solution. The citral to β -cyclodextrin mole ratio was 1.5. The amount of citral added on was 0.4 kg of citral per air-dried metric ton ("ADMT") of fluff. Fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials Jesup, GA. The calculated increase in moisture content of the substrate was 0.1 wt%. Because the moisture increase was less than about 1 wt% or less, no drying step was required. The overall odor score was +52 as determined by the blind, triangular forced-choice test method with an expert panel discussed above. At an add-on of only 0.4 kg citral /ADMT fluff pulp, this sample provided odor control performance as good as that for a sample made with an add on of 2.0 kg citral /ADMT fluff pulp (i.e. Example 7). Specifications and odor score measurements for Example 8 are given in TABLE 18 and TABLE 19.

[0137] [TABLE 18]

SPECIFICATION												
Sample	Description	Solution					Solution Add-on (g soln)	Solids Add-On (g solids)	CD Add-On (kg CD)	Citral Add-On (kg Citral)	Glycerol Add-On (kg glycerol /ADMT fluff)	Fluff Moisture Increase
		Component	Liquid Conc	Soln Wt. Fraction	T (°C)	pH						
Example	β-CD/Citral complex	glycerol	0.85	0.666	86	12.5	0.011	0.002	2.0	0.40	7.4	0.1%
		water	0.15	0.118								
		Subtotal		0.784								
		β-Cyclodextrin		0.180								
		Citral		0.036								
	Total		1.00									
		Mole Ratio (mol CD/mol Citral) =					1.5					
		Solution Preparation:										
	MW (g/mol)		g used	%								
	1135	CD	11.5	18.0%	21.6%		Total	13.8				
	152.2	Citral	2.31	3.6%								
		Soln	50.0	78.4%								
		Total	63.8	100%								

[0138] [TABLE 19]

ODOR EVALUATION									
Triangular Forced-Choice Odor Evaluation			time (min)	Liquid Dosing Level				Odor Score	
Test	2 Controls	1 Unknown		2.5	5	10			
1	Rayfloc/water	OCF/urine	10	6±	6±	6±		+18	+52
			100	6±	6±	6-		+6	
2	Rayfloc/urine	OCF/urine	10	6+	6+	2+		+14	
			100	2+	6+	6+		+14	

[0139] Example 9: Substrate Preparation

[0140] A sample was prepared using a 100% water (i.e. a 0/100 ratio of glycerol/water) and a pH adjustment of 12.5 using β -cyclodextrin from Acros Organics and citral from Perfumer's Apprentice. The solution was mixed and applied at a temperature of 86° C. At the application temperature of 86° C, the components formed a relatively stable, milky-white dispersion. The citral to β -cyclodextrin mole ratio was 1.5. The amount of citral add-on was 0.4 kg of citral per air-dried metric ton ("ADMT") of fluff. Fluff pulp in sheet form was an untreated southern pine Kraft pulp sold under the trade name RAYFLOC® JLDE that was obtained from Rayonier Advanced Materials Jesup, GA. The calculated increase in moisture content of the substrate was 1.1 wt%. Because the moisture increase was about 1 wt%, no drying step was required. The overall odor score was +36 as determined by the blind, triangular forced-choice test method with an expert panel discussed above. The odor control performance of this reduced-cost, 100% water solution sample was good -- almost as good as the sample made in Example 8 with the 85/15 glycerol/water solution. The mixture formed a dispersion after the addition of the citral and formation of the complex at 86° C., but when the concentration of cyclodextrin and citral in the solution was less than about 20 wt%, the size of the precipitated particles was very small, odor control performance was good and the dispersion could be sprayed easily onto the substrate. Specifications and odor score measurements for Example 10 are given in TABLE 20 and TABLE 21.

[0141] [TABLE 20]

SPECIFICATION												
Sample	Description	Solution					Solution Add-on (g soln /g fluff)	Solids Add-On (g solids /g fluff)	CD Add-On (kg CD /ADMT fluff)	Citral Add-On (kg Citral /ADMT fluff)	Glycerol Add-On (kg glycerol /ADMT fluff)	Fluff Moisture Increase (%)
		Component	Liquid Conc	Soln Wt. Fraction	T (°C)	pH						
Example	β-CD/Citral complex	glycerol	0.00	0.000	86	12.5	0.013	0.002	2.0	0.40	0.0	1.1%
		water	1.00	0.822								
		Subtotal:		0.822								
		β-Cyclodextrin		0.148								
		Citral		0.030								
		Total:		1.00								
		Mole Ratio (mol CD/mol Citral) =										
		Solution Preparation:										
	MW (g/mol)		g used	%								
	1135	CD	9.0	14.8%	17.8%		Total	10.8				
	152.2	Citral	1.81	3.0%								
		Soln	50.0	82.2%								

[0142] [TABLE 21]

ODOR EVALUATION										
Triangular Forced-Choice Odor Evaluation			time (min)	Liquid Dosing Level (g liq / g fluff)				Odor Score Summary		
Test	2 Controls	1 Unknown		2.5	5	10				
1	Rayfloc/water	OCF/urine	10	6±	6±	6-		+6	0	+36
			100	6±	6-	6-		-6		
2	Rayfloc/urine	OCF/urine	10	6+	6+	6+		+18	+36	
			100	6+	6+	6+		+18		

5 [0143] CONCLUSION AND SUMMARY OF EXAMPLES 5-9

[0144] A summary of Examples 5 to 9 is given in TABLE 22 below. At a glycerol/water ratio of 85/15, and citral add on in the range of 2.0 – 2.4 kg citral /ADMT fluff pulp, an increase in the citral:cyclodextrin mole ratio from 1:1 to 1.5:1 increased the odor control performance (as indicated by increasing TOTAL ODOR SCORE) by about as much as an increase in solution pH from 9.6 to 12.5. The unadjusted pH of the solution was about 6.5. Using a lower add-on of the same solution to provide a reduced citral add-on of 0.4 kg citral /ADMT fluff pulp provided about the same level of very good performance (i.e. +52), again at a citral:cyclodextrin mole ratio of 1.5:1 and a pH of 12.5. Examples 5 to 8 had clear, water-white solutions at the mixing

and application temperature of 86° C. The complex precipitated on the surface of the cellulose fibers as the solution and treated fibers cooled to room temperature.

[0145] [TABLE 22]

Example	Glycerol/ Water Ratio	Citral:CD Mole Ratio	Citral/CD Complex Solution Conc. (wt%)	Citral Add-on (kg Citral /ADMT fluff pulp)	pH	Mixing & Application Temp (°C)	Moisture Increase of Substrate (wt%)	Total Odor Control Score vs. Rayfloc fluff pulp
5	85/15	1 : 1	22.6%	2.4	9.6	86° C.	1.0%	-28
6	85/15	1 : 1	24.9%	2.4	12.5	86° C.	0.9%	+16
7	85/15	1.5 : 1	21.6%	2.0	12.5	86° C.	0.6%	+56
8	85/15	1.5 : 1	21.6%	0.4	12.5	86° C.	0.1%	+52
9	0/100	1.5 : 1	17.8%	0.4	12.5	86° C.	1.1%	+36

5 [0146] It was an unexpected discovery to learn that the odor control performance of a β -cyclodextrin / citral complex could be greatly improved by forming the complex in a glycerol/water solution at a citral: β -cyclodextrin mole ratio of about 1.5:1 and at an elevated pH of about 12.5.

10 [0147] The exact physical dimensions and morphology of the cyclodextrin complex that precipitated *in situ* on the solid fiber surface have not been characterized. However, without being bound to a singular theory, the inventors believe that the particle size of the complex precipitated in solution, as the solution cools to room temperature, is a measure of the morphology of the complex on a substrate surface. The mean size of solid particles of the complex precipitated from a solution at an elevated temperature at pH 12.5 is in the range of 50
15 μm or less. A solution pH of about 12.5 or greater should be used for good odor control performance. Odor control performance is poorer when a solution pH of 9.6 or less is used to treat a substrate. The mean size of solid particles of complex precipitated from a solution at an elevated temperature at pH 9.6 or less is in the range of 100 – 400 μm . The particles size is believed to be controlled by the pH of the solution, *inter alia*.

20 [0148] The above discussed results were unanticipated. There is no known discussion of the effect of pH on the odor control performance of cyclodextrin complexes in the prior art. The effect may be consistent with the inventors' additional discovery that the complex precipitates

into very fine particles when it is precipitated from a solution with a pH of around 12.5. Odor control performance of a complex deposited from solution may be related to its solid phase morphology on the substrate surface. Hydroxyl groups on the outer surface of a cyclodextrin molecule start to deprotonate at about pH 12 (i.e. pKa range about 12.1 – 13.5). It is known that cyclodextrins and their complexes undergo self-assembly in solution to form aggregate structures. This aggregate formation might be affected by a surface charge related to deprotonation, and in turn, affect the size of precipitated particles and the morphology of complexes deposited on a substrate surface. Others, such as in U.S. Pat. No. 5,429,628, have claimed that small cyclodextrin particles improve the release and speed of release of active. Particles of less than about 12 microns were preferred for providing a quick release of active when the complexes were wetted. U.S. Pat. No. 5,429,628, however, achieved this reduction in particle size via grinding and pulverizing in a fluid energy mill, or by agitation or sonication. The inventors are unaware of the use of solution pH to adjust the particle size of cyclodextrin complexes, as disclosed herein.

[0149] The inventors also unexpectedly discovered that the odor control performance can be sensitive to the molar ratio of citral to cyclodextrin in the complex. U.S. Pat. No. 5,429,628 and WO 98/26808 indicate that, in general, perfume/cyclodextrin complexes have a molar ratio of perfume to cyclodextrin of about 1:1, although sometimes greater, but typically the loading of perfume to cyclodextrin is about 7 wt% to 12 wt%. There are practical limitations to the loading of perfume in cyclodextrin complexes but the inventors are unaware of any disclosure about how loading affects odor control performance. For citral/ β -cyclodextrin complexes, a molar ratio of citral to β -cyclodextrin of 1:1 would correspond to a loading of 11.8 wt%, as generally disclosed by U.S. Pat. No. 5,429,628. However, the molar ratio of citral to β -cyclodextrin of 1.5:1 would correspond to a loading of 16.7 wt%. Taking into account a moisture content of β -cyclodextrin of 10 wt%, the molar ratio of citral to dry β -cyclodextrin of 1.5:1 would correspond to a loading of 18.3 wt%. That is, such ranges of citral loading are well outside the range taught in U.S. Pat. No. 5,429,628. The inventors are unaware of a technical explanation for the disclosed higher range of citral loading, which may be facilitated by the high solution pH in which the complex is formed. Even though it may be possible that the actual quantitative values could be different for the loading of different essential oils, the inventors expect that the requirements for achieving the

excellent odor control include a minimum loading value of essential oil to cyclodextrin and a solution pH above about 12.0, as a common feature of these systems.

[0150] The complexes in Examples 5 to 9 formed a clear, homogeneous solution at an elevated temperature of about 86° C. This step did not improve the odor control performance of the complex. However, it greatly improved the ability to handle the solution and uniformly apply it to a substrate surface. A solution of a β -cyclodextrin / citral complex sprayed on fluff pulp at an elevated temperature of about 86° C. would precipitate *in situ* on the fiber surface as it cooled to room temperature. Solution concentrations of the citral/cyclodextrin complexes were in the range of 21.6 wt% - 24.9 wt%. The solution concentration of the complex can reduce the solution add-on to keep the moisture increase of the substrate below about 1 wt%. However, the ratio of odor-controlling compound to cyclodextrin is also supportive for imparting good odor control performance to a substrate. For citral, the optimum molar ratio of citral to β -cyclodextrin is about 1.5-1.75 moles of citral to 1.0 mole of β -cyclodextrin. At molar ratios of about 1:1 and less, odor control performance is reduced (at equal add-ons of citral to substrate). At molar ratios of 1.75:1 and greater, volatile, uncomplexed citral imparts unnecessary color and scent to the substrate. Volatile, and costly, scented essential oils used in absorbent products tend to be lost prematurely. It is desirable to have the essential oils contained in a stable complex and released only when in contact with aqueous body fluids.

[0151] The ratio of glycerol/water also plays a role in the solution chemistry discussed above. For example, samples made with higher ratios of water (e.g., 50% glycerol/50% water) do not perform as well as those made with the 85% glycerol/15% water ratio, even when they are made at the preferred 1.5 moles citral:mole cyclodextrin and pH 12.5.

[0152] However, it is noteworthy that Example 9 provided almost as good odor control performance using a 100% water solution and was capable of delivering 0.4 kg citral /ADMT fluff pulp with a moisture increase of only 1.1%. This unexpected, practical discovery provides good odor control performance at much reduced raw material cost. In this embodiment, for good odor control performance from a 100% water solution, conditions must be right for the formation of a fine precipitate upon the addition of citral to a β -cyclodextrin/water solution. A solution concentration at 86° C. and pH 12.5 for the complex is lower -- in the range of about 18wt.% or

less. At higher concentrations, an undesirably coarse precipitate forms and odor control performance deteriorates.

[0153] In the foregoing description, the solutions containing cyclodextrins for imparting odor control to materials used in absorbent products and associated products and methods of the present application have been described with reference to specific embodiments thereof. It will, however, be evident that various modifications and changes may be made thereto without departing from the broader spirit and scope of the invention. The specification and drawings are, accordingly, to be regarded in an illustrative rather than a restrictive sense. Throughout this specification, unless the context requires otherwise, the word “comprise” and its variations, such as “comprises” and “comprising,” will be understood to imply the inclusion of a stated item, element or step or group of items, elements or steps, but not the exclusion of any other item, element or step or group of items, elements or steps. Furthermore, the indefinite article “a” or “an” is meant to indicate one or more of the item, element, or step modified by the article.

CLAIMS

1. A substrate comprising an odor-controlling solution applied thereupon,
wherein the odor-controlling solution comprises:
a polar organic solvent,
5 water,
a cyclodextrin, and
an odor-controlling compound, and
wherein the substrate comprises at least 0.2 kg of the odor-controlling compound per
1000 kg of the substrate.

2. The substrate according to claim 1, wherein the substrate has an increase in moisture
content of less than 1 wt. % as a result of the odor controlling solution having been applied
thereupon.

3. The substrate according to claim 1, wherein the substrate is
a fluff pulp in the form of a compressed sheet or an air-laid fiber form,
a synthetic or cellulosic fiber spun-bond or carded nonwoven, woven fabric, or solid
material, or
an absorbent article.

4. The substrate according to claim 3,
wherein the substrate is the solid material, and
wherein the solid material is selected from the group consisting of a polymeric foam,
leather, and plastic film.

5. The substrate according to claim 3,
wherein the substrate material is the absorbent article, and
wherein the absorbent article is an adult incontinence control pad, feminine hygiene pad,
pull-up underwear, adult brief, baby diaper, or bed pad.

6. The substrate according to claim 1, wherein the polar organic solvent is selected from the group consisting of glycerol, propylene glycol, polypropylene glycol, polyethylene glycol, and mixtures thereof.
- 5 7. The substrate according to claim 1, wherein the cyclodextrin is an unsubstituted or substituted α -, β - or γ -cyclodextrin.
8. The substrate according to claim 1, wherein the odor-controlling compound is at least one of
- 10 an essential oil is selected from the group consisting of citral, limonene, octanol, lavandin, geranium, grapefruit, linalool, citronellol, linalyl acetate, geraniol, eugenol, floracetate, cardamom, eucalyptus, juniper berry, lavender, lemon verbena, lemongrass, patchouli, rose, spearmint, tea tree, oregano, persimmon and mixtures thereof,
- a botanical extract,
- 15 an odor-controlling substance selected from the group consisting of benzaldehyde, cinnamic aldehyde, ligustral, florhydral, and mixtures thereof, or
- an antimicrobial substance selected from the group consisting eucalyptus, lavender, thyme, peppermint, cajuput, cinnamon, clove, sage, tea tree oils, and mixture thereof.
- 20 9. The substrate according to claim 1, wherein the odor-controlling solution comprises 1.5 to 1.75 moles of the odor controlling compound per mole of the cyclodextrin.
10. The substrate according to claim 1, wherein the odor-controlling solution has a pH greater than or equal to 12.0.
- 25 11. The substrate according to claim 1,
- wherein the odor-controlling solution comprises:
- 67 wt. % of glycerol as the polar organic solvent,
- 12 wt. % of the water, and
- 30 21 wt. % of the cyclodextrin and the odor controlling compound,

wherein the cyclodextrin is unsubstituted β -cyclodextrin, and

wherein the odor-controlling compound is citral,

wherein the citral and the unsubstituted β -cyclodextrin are present in a mole ratio of 1.5 moles of the citral per mole of the unsubstituted β -cyclodextrin,

5 wherein the substrate is fluff pulp,

wherein the substrate comprises 0.4 kg of the citral per 1000 kg of the fluff pulp, and

wherein the substrate has an increase in moisture content of less than 0.13 wt. % as a result of the odor controlling solution having been applied thereupon.

10 13. An article comprising the substrate according to claim 1.

14. A method of preparing an odor-controlling substrate, comprising:

a) providing a substrate,

b) applying an odor-controlling solution onto the substrate to obtain the odor-

15 controlling substrate,

wherein the odor-controlling solution comprises:

a polar organic solvent,

water,

a cyclodextrin, and

20 an odor-controlling compound, and

wherein, after step b), the odor controlling substrate comprises at least 0.2 kg of the odor controlling compound per 1000 kg of the odor controlling substrate.

15. The method according to claim 14, wherein applying the odor-controlling solution onto
25 the substrate in step b) increases moisture content of the substrate by less than 1 wt.%.

16. The method according to claim 14, wherein the substrate is

a fluff pulp in the form of a compressed sheet or an air-laid fiber form,

a synthetic or cellulosic fiber spun-bond or carded nonwoven, woven fabric, or solid

30 material, or

an absorbent article.

17. The method according to claim 14, wherein the polar organic solvent is selected from the group consisting of glycerol, propylene glycol, polypropylene glycol, polyethylene glycol, and mixtures thereof.

18. The method according to claim 14, wherein the cyclodextrin is an unsubstituted or substituted α -, β - or γ -cyclodextrin.

19. The method according to claim 14, wherein the odor-controlling solution comprises 1.5 to 1.75 moles of the odor controlling compound per mole of the cyclodextrin.

20. The method according to claim 14, wherein the odor-controlling solution has a pH greater than or equal to 12.0.

21. The method according to claim 14,
wherein the odor-controlling solution comprises:

67 wt. % of glycerol as the polar organic solvent,

12 wt. % of the water, and

21 wt. % of the cyclodextrin and the odor-controlling compound,

wherein the cyclodextrin is unsubstituted β -cyclodextrin, and

wherein the odor-controlling compound is citral,

wherein the citral and the unsubstituted β -cyclodextrin are present in a mole ratio of 1.5 moles of the citral per mole of the unsubstituted β -cyclodextrin,

wherein the substrate is fluff pulp,

wherein the substrate comprises 0.4 kg of the citral per 1000 kg of the fluff pulp, and

wherein the substrate has an increase in moisture content of less than 0.13 wt. % as a result of the odor controlling solution having been applied thereupon.

22. The method according to claim 21, wherein step b) comprises applying 0.011 g of the odor controlling solution per g of the substrate material at a solution temperature of 86° C and pH 12.5,

wherein the substrate material is a sheet of fluff pulp.

5

23. A substrate comprising an odor-controlling solution applied thereupon,

wherein the odor-controlling solution comprises:

water,

an optional polar organic solvent,

10

a cyclodextrin, and

an odor-controlling compound, and

wherein the substrate comprises at least 0.2 kg of the odor-controlling compound per 1000 kg of the substrate, and

15

wherein the odor-controlling solution comprises 1.5 to 1.75 moles of the odor-controlling compound per mole of the cyclodextrin, and

wherein the odor-controlling solution has a pH greater than pH 12.0, and

wherein the concentration of cyclodextrin and odor-control compound in the solution is about 18 wt%, and

20

wherein the substrate has an increase in moisture content of about 1.1 wt% as a result of the odor controlling solution having been applied thereupon.

25

ABSTRACT

Solutions containing cyclodextrins for imparting odor control to materials used in absorbent products and associated products and methods. The solutions containing cyclodextrins for imparting odor control to materials used in absorbent products contain an optional polar
5 organic solvent, water, cyclodextrin, and one or more odor-controlling compounds. The solutions can be used to uniformly apply a homogeneous solution of a cyclodextrin complex onto a solid surface whereby the cyclodextrin complex precipitates *in situ* on the solid surface. The cyclodextrin complex precipitates deposited from these solutions rapidly release odor-masking scents when hydrated with urine and can be deposited on substrates more effectively than
10 cyclodextrin complexes in solid form.

FIG. 1

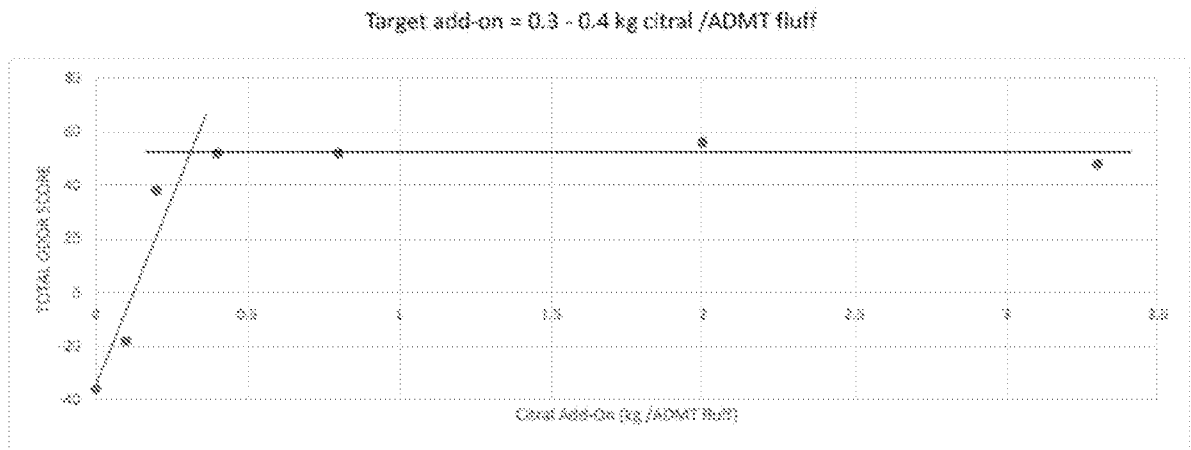


FIG. 2

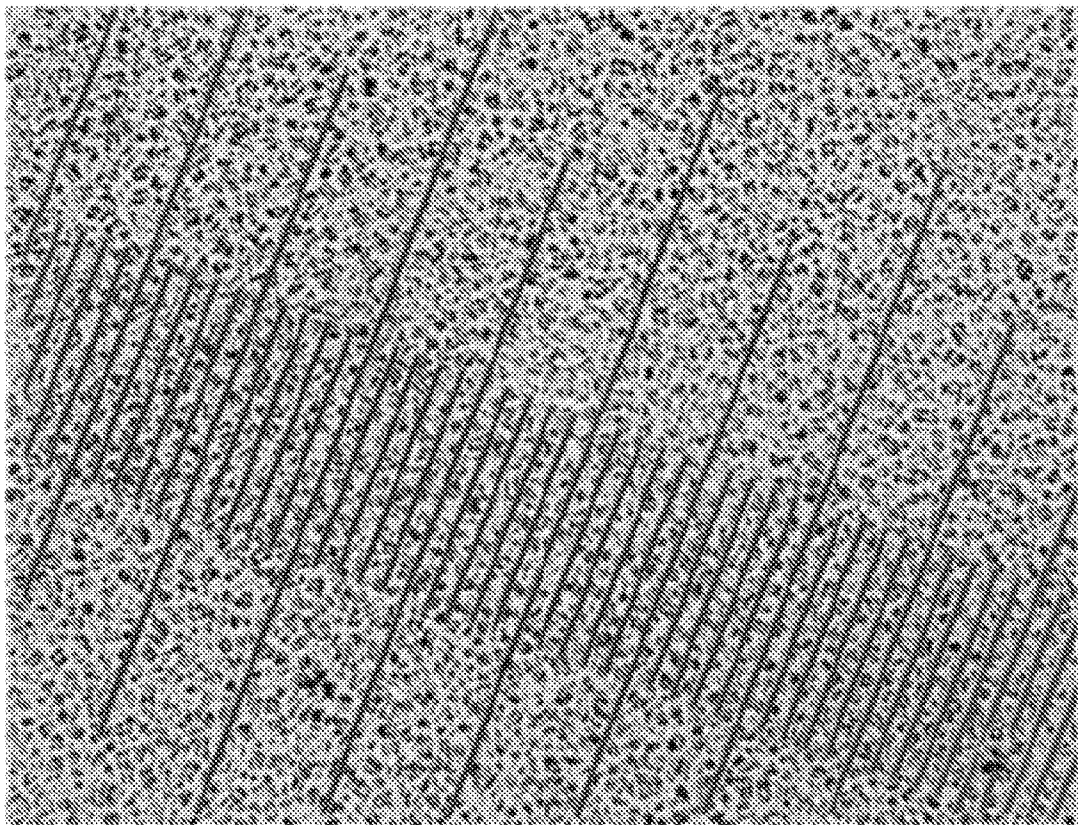
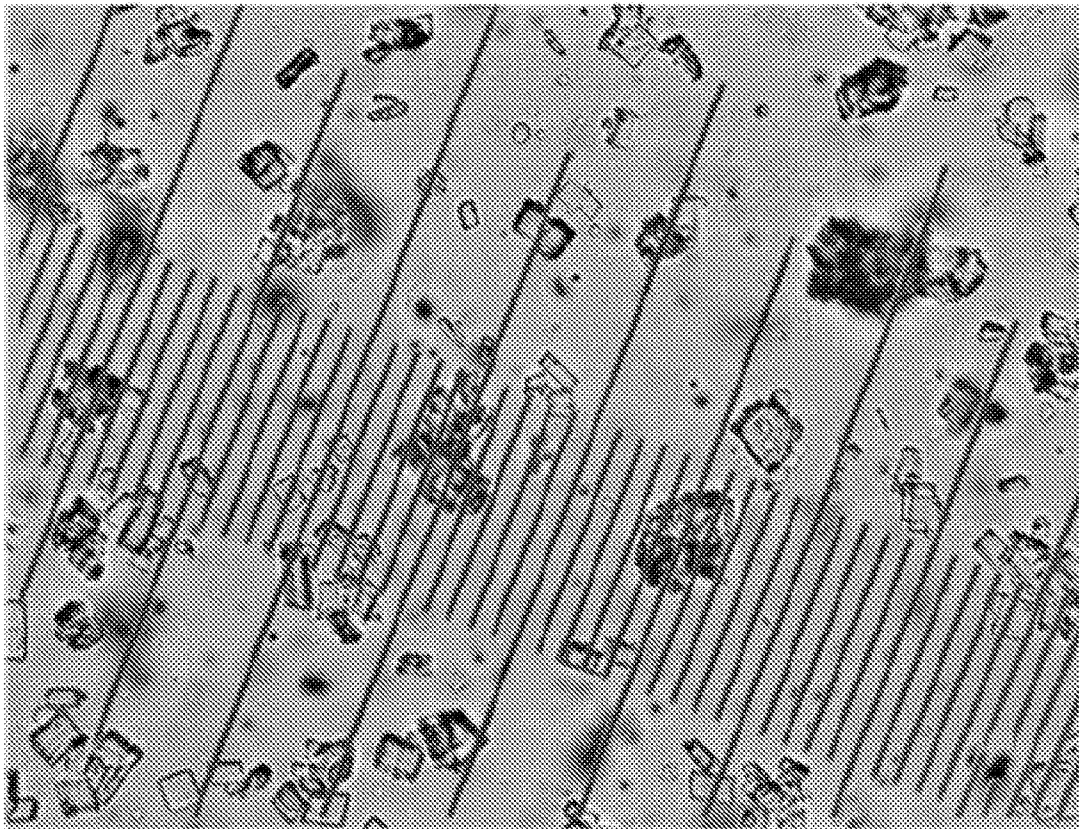


FIG. 3



Electronic Acknowledgement Receipt

EFS ID:	45506292
Application Number:	63332443
International Application Number:	
Confirmation Number:	4742
Title of Invention:	SUBSTRATES TREATED WITH SOLUTIONS CONTAINING CYCLODEXTRINS FOR IMPARTING ODOR CONTROL TO MATERIALS USED IN ABSORBENT PRODUCTS
First Named Inventor/Applicant Name:	Harry CHMIELEWSKI
Customer Number:	513
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