

IN THE UNITED STATES DISTRICT COURT  
NORTHERN DISTRICT OF ILLINOIS  
EASTERN DIVISION

PRESERVATION PRODUCTS, LLC, )  
an Indiana limited liability company, )

Plaintiff, )

vs. )

NUTRACEUTICAL CLINICAL )  
LABORATORIES )

INTERNATIONAL, INC., a Florida )  
corporation; and NATURAL )  
PRESERVATION TECHNOLOGY, )  
INC., a Florida corporation; and PAUL )  
SIMMONS; )

Defendants )

No. **01C 7403**

JUDGE ANDERSEN

MAGISTRATE JUDGE DENLOW

*Denlow*  
U.S. DISTRICT COURT

01 SEP 25 PM 1:50

FILED-ED4

**DOCKETED**  
SEP 26 2001

COMPLAINT

Plaintiff, Preservation Products, LLC (hereinafter "Plaintiff") by its attorneys bring this complaint against Defendants, Nutraceutical Clinical Laboratories International, Inc. (hereinafter "NCLII") and Natural Preservation Technology, Inc. (hereinafter "NPTI") and Paul Simmons and states as follows:

Nature of Action

1. This is an action for preliminary and permanent injunctive relief, for compensatory and punitive damages and for attorneys fees and costs incurred in bringing this action based upon defendants' patent infringement, trademark infringement, unfair competition and deceptive practices in the marketing, manufacturing, offering for sale, sale and use of the patented product "LIQUISORB" and wax cellulose technology. The defendants' acts are in violation of the United

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States patent laws, (35 USC §§1-376), the Trademark Act of the United States (15 USC §1051 et seq.) and the Uniform Deceptive Trade Practices Act (815 ILCS 510/2).

### **JURISDICTION AND VENUE**

2. This Court has jurisdiction over this action pursuant to 28 USC §§1331 and 1338 and 15 USC §1121. This court also has jurisdiction under 28 USC §1332, in that the matter in controversy is between citizens of different states and exceeds the sum or value of \$75,000.00 exclusive of interest and costs. This action is authorized and instituted pursuant to 35 USC §271, 15 USC §1125 and 815 ILCS 510/3.

3. Venue is proper in this Court pursuant to 28 USC §1391(b) and (c) as the defendants are conducting business within the boundaries of this judicial district including the offering for sale of products bearing a trademark which are the subjects of this action and the unlawful acts and their effects are occurring and will continue to occur within the boundaries of this judicial district.

### **PARTIES**

4. The Plaintiff is an Indiana LLC registered to do business and doing business in Illinois with its office located at 6234 North Olcott, Chicago, Illinois. It is the assignee-owner of several patents for wax cellulose technology products which includes a natural preservative known as LIQUISORB. The Plaintiff is the owner of the trademark "LIQUISORB".

5. NCLII is a Florida corporation that trades its stock over-the-counter under the call letters NCCL. NCLII via its interactive website and its shareholder newsletter claims, in this judicial district and elsewhere, that it is the manufacturer and owner of the natural preservative, LIQUISORB, and NCLII is offering via its website to provide the preservative to others.

6. NPTI, according to NCLII's website is a wholly owned subsidiary of NCLII and is the manufacturer of NCLII's natural preservative which is the subject of this action.

7. Defendant, Paul L. Simmons, is an individual who, on information and belief, resides in or near St. Petersburg, Florida. Mr. Simmons is the president, chief executive officer, founder and chairman of Nutraceutical Laboratories International, Inc. (NCLII) and Natural Preservation Technology Inc. (NPTI). Mr. Simmons is and has been personally involved in the design, manufacture, marketing, development and sale of the product and processes herein charged with patent infringement, unfair competition under Section 1051 et seq. of the Lanham Act and deceptive trade practices in violation of the Uniform Deceptive Trade Practices Act. As described subsequently herein, Mr. Simmons is also the president, chief executive officer, founder and chairman of another corporation (Nutraceutical Clinical Laboratories, Inc. [NCLI]) which was a former licensee of Plaintiff under certain of the patents involved in this action.

**STATEMENT OF CLAIMS**  
**COUNT I**

**INFRINGEMENT OF U.S. PATENT 5,840,249**

8. This Count is for patent infringement under the patent laws of the United States (35 USC §§1-376). Plaintiff incorporates by reference the allegations of ¶¶1-7 above.

9. On November 24, 1998, United States Patent No. 5,840,249 ("the '249 patent"), entitled PRESERVATIVE FOR ORGANIC MATERIALS, was duly and legally issued. A true copy of the '249 patent is attached to this Complaint as Exhibit A and is incorporated herein by reference.

10. On April 29, 1999, the '249 patent was assigned to Plaintiff. This assignment was

duly recorded and a true copy of the assignment is attached to this Complaint as Exhibit B and is incorporated herein by reference.

11. On information and belief, NCLII is and has been infringing or inducing or contributing to the infringement of the '249 patent by making, using, marketing, offering for sale, and selling products made by processes covered by claims of the '249 patent. NCLII will continue to infringe or induce or contribute to the infringement of the '249 patent unless enjoined by this Court.

12. On information and belief, defendant NPTI is and has been infringing the '249 patent by making and using products made by processes covered by claims of the '249 patent. NPTI will continue to infringe the '249 patent unless enjoined by this Court.

13. On information and belief, defendant Paul Simmons is and has been infringing the '249 patent through his personal involvement in the design, manufacture, marketing, use, sale and offer for sale of products made by processes covered by claims of the '249 patent and by contributing to and inducing others to infringe the '249 patent by using the inventions claimed in those patents. Mr. Simmons will continue to infringe the '249 patent unless enjoined by this court.

14. Defendants have received notice that they do not have the right to manufacture, market, sell, sub-license, or distribute products made by the processes of the '249 patent, but have willfully continued their infringement thereof.

15. By reason of defendants' aforementioned acts of infringement, Plaintiff has been and will continue to be irreparably damaged unless the defendants are enjoined from infringing the '249 patent.

**COUNT II**

**INFRINGEMENT OF U.S. PATENT 6,103,294**

16. This Count is for patent infringement under the patent laws of the United States (35 USC §§1-376). Plaintiff incorporates by reference the allegations of ¶¶1-7 above.

17. On August 15, 2000, United States Patent No. 6,103,294 (“the ‘294 patent”), entitled PRESERVATIVE FOR DIGESTIBLE FOOD AND BEVERAGE PRODUCTS, was duly and legally issued. A true copy of the ‘294 patent is attached to this Complaint as Exhibit C and is incorporated herein by reference.

18. On April 29, 1999, the corresponding application for the ‘294 patent was assigned to Plaintiff. This assignment was duly recorded and a true copy of the assignment is attached to this Complaint as Exhibit D and is incorporated herein by reference.

19. On information and belief, NCLII is and has been infringing or inducing or contributing to the infringement of the ‘294 patent by making, using, offering for sale, and selling products covered by claims of the ‘294 patent. NCLII will continue to infringe or induce or contribute to the infringement of the ‘294 patent unless enjoined by this Court.

20. On information and belief, defendant NPTI is and has been infringing the ‘294 patent by making and using products made by processes covered by claims of the ‘294 patent. NPTI will continue to infringe the ‘294 patent unless enjoined by this Court.

21. On information and belief, defendant Paul Simmons is and has been infringing the ‘294 patent through his personal involvement in the design, manufacture, marketing, use, sale and offer for sale of products covered by claims of the ‘294 patent and by contributing to and inducing others to infringe the ‘294 patent by using the inventions claimed in those patents. Mr.

Simmons will continue to infringe the '294 patent unless enjoined by this court.

22. Defendants have received notice that they do not have the right to manufacture, market, sell, sub-license, or distribute products made by the processes of the '294 patent, but have willfully continued their infringement thereof.

23. By reason of the defendants' aforementioned acts of infringement, Plaintiff has been and will continue to be irreparably damaged unless the defendants are is enjoined from infringing the '294 patent.

### **COUNT III**

#### **INFRINGEMENT OF U.S. PATENT 6,171,550**

24. This Count is for patent infringement under the patent laws of the United States (35 U.S.C. §§1-376). Plaintiff incorporates by reference the allegations of ¶¶1-7 as if fully set forth herein.

25. On January 9, 2001, United States Patent No. 6,171,550 ("the '550 patent"), entitled NON-TOXIC BASE INGREDIENT FOR CONSUMER PRODUCTS, was duly and legally issued. A true copy of the '550 patent is attached to this Complaint as Exhibit E and is incorporated herein by reference.

26. On April 29, 1999, the correspondence application for the '550 patent was assigned to Plaintiff. This assignment was duly recorded and a true copy of the assignment is attached to this Complaint as Exhibit F and is incorporated herein by reference.

27. On information and belief, NCLII is and has been infringing or inducing or contributing to the infringement of the '550 patent by making, using, offering for sale, and selling

products made by processes covered by claims of the '550 patent. NCLII will continue to infringe or induce or contribute to the infringement of the '550 patent unless enjoined by this Court.

28. On information and belief, NPTI is and has been infringing the '550 patent by making and using products made by processes covered by claims of the '550 patent. NPTI will continue to infringe the '550 patent unless enjoined by this Court.

29. On information and belief, defendant Simmons is and has been infringing the '550 patent through his personal involvement in the design, manufacture, marketing, use, sale and offer for sale of products made by processes covered by claims of the '550 patent and by contributing to and inducing others to infringe the '550 patent by using the inventions claimed in those patents. Mr. Simmons will continue to infringe the '550 patent unless enjoined by this court.

30. Defendants have received noticed that they do not have the right to manufacture, market, sell, sub-license or distribute products made by the processes of the '550 patent, but have willfully continued their infringement thereof.

31. By reason of defendants' aforementioned acts of infringement, Plaintiff has been and will continue to be irreparably damaged unless Defendants are enjoined from infringing the '550 patent.

#### **COUNT IV**

#### **INFRINGEMENT OF U.S. PATENT 5,412,090**

32. This Count is for patent infringement under the patent laws of the United States

(35 U.S.C. §§1-376). Plaintiff incorporates by reference the allegations of ¶¶1-7 as if fully set forth herein.

33. On May 2, 1995, United States Patent No. 5,412,090 (“the ‘090 patent”), entitled HYDROUS CELLULOSE PULP FOR NON-PAPER PRODUCTS, was duly and legally issued. A true copy of the ‘090 patent is attached to this Complaint as Exhibit G and is incorporated herein by reference.

34. On April 29, 1999, the corresponding application for the ‘090 patent was assigned to Plaintiff. This assignment was duly recorded and a true copy of the assignment is attached to this Complaint as Exhibit H and is incorporated herein by reference.

35. On information and belief, NCLII is and has been infringing, or inducing or contributing to the infringement of, the ‘090 patent by making, using, offering for sale, and selling products covered by claims of the ‘090 patent. NCLII will continue to infringe, or induce or contribute to the infringement of, the ‘090 patent unless enjoined by this Court.

36. On information and belief, NPTI is and has been infringing the ‘550 patent by making and using products made by processes covered by claims of the ‘550 patent. NPTI will continue to infringe the ‘550 patent unless enjoined by this Court.

37. On information and belief, defendant Simmons is and has been infringing the ‘090 patent through his personal involvement in the design, manufacture, marketing, use, sale and offer for sale of products covered by claims of the ‘090 patent and by contributing to and inducing others to infringe the ‘090 patent by using the inventions claimed in those patents. Mr. Simmons will continue to infringe the ‘090 patent unless enjoined by this Court.

38. Defendants have received notice that they do not have the right to manufacture,



market, sell, sub-license or distribute products made by the processes of the '090 patent, but have willfully continued their infringement thereof.

39. By reason of defendants' aforementioned acts of infringement, Plaintiff has been and will continue to be irreparably damaged unless defendants are enjoined from infringing the '090 patent.

### COUNT V

#### **LANHAM ACT (15 USC 1051 et seq.)**

40. This Count is brought under the Trademark Act of the United States (15 USC 1051 et seq.). Plaintiff incorporates by reference the allegations of ¶¶1-7 as if fully set forth herein.

41. On September 29, 1999, NCLI, but not NCLII or NPTI, received a license from Plaintiff which provided, among other things, a limited right to test, manufacture and market a food grade preservative known as LIQUISORB to the soft drink and food industries, which license was terminated on February 3, 2001.

42. Plaintiff has never granted a license or other right to NCLII or NPTI or Paul Simmons to manufacture, market, use, sell, offer for sale the natural preservative LIQUISORB or otherwise use the term LIQUISORB. Without Plaintiff's authority and some time after Plaintiff began using the trademark LIQUISORB, NCLII began to use and is continuing to use the trademark LIQUISORB in interstate commerce on its interactive website, and in its shareholder newsletter and, on information and belief, elsewhere as a trademark for a preservative for food and beverages. Moreover, NCLII falsely described LIQUISORB as "our product". A true copy of relevant portions of the NCLII website and shareholder newsletter is attached to this

Complaint respectively as Exhibits I and J, and is incorporated herein by reference.

43. Without Plaintiff's authority and some time after Plaintiff began using the trademark LIQUISORB, NPTI began to use and is continuing to use the trademark LIQUISORB in interstate commerce via the interactive website of NCLII as a trademark for a preservative for food and beverages.

44. Without Plaintiff's authority and after Plaintiff began using the trademark LIQUISORB, Paul Simmons through his personal involvement in the marketing of LIQUISORB, by NCLII and NPTI, began to use and is continuing to use the trademark LIQUISORB as a trademark for a preservative for food and beverages.

45. The defendants are falsely and misleadingly using the trademark LIQUISORB in a manner which is likely to cause confusion or mistake, or to deceive as to the affiliation, connection, or association of NCLII, NPTI and Paul Simmons with Plaintiff, or as to the origin, sponsorship, or approval of the goods, services or commercial activities of NCLII, NPTI and Paul Simmons. Further, the defendants' use of the term LIQUISORB is commercial activity or promotion that misrepresents the nature, characteristics, or qualities of the goods, services and commercial activities of NCLII, NPTI and Paul Simmons.

46. As a result of the foregoing, Plaintiff has suffered and will continue to suffer irreparable damages, and NCLII, NPTI and Paul Simmons have been unjustly enriched thereby, in an amount currently undetermined and for which there is no adequate remedy at law.

**COUNT VI**

**DECEPTIVE TRADE PRACTICES**

47. This Count is brought under the Uniform Deceptive Trade Practices Act (815 ILCS 510/1 et seq.). Plaintiff incorporates ¶¶1-7 and ¶¶41-44 as if fully set forth herein.

48. Defendant NCLII has and continues to willfully engage in deceptive conduct which creates a likelihood of confusion or misunderstanding, in that NCLII via its interactive website and its shareholder newsletter falsely represents to consumers, the investing public and others that:

- a) NCLII lawfully manufactures all-natural liquid preservatives under the trademark of LIQUISORB when in fact, NCLII is either not manufacturing such a preservative at all or is not lawfully manufacturing such a preservative;
- b) LIQUISORB is a NCLII product;

49. Defendant NPTI has and continues to wilfully engage in deceptive conduct which creates a likelihood of confusion and misunderstanding, in that NPTI via its parent company's website falsely represents to consumers, the investing public and other businesses that NPTI is the lawful manufacturer of an all-natural liquid preservative for the food and beverage industry, when in fact NPTI is either not manufacturing such a preservative at all or is not lawfully manufacturing such a preservative.

50. Defendant Paul Simmons through his personal involvement in the marketing of LIQUISORB by NCLII and NPTI falsely represents to the public that NCLII and NPTI are lawfully manufacturing an all-natural liquid preservative, LIQUISORB, when in fact NCLII and

NPTI are either not manufacturing such a preservative or are not lawfully manufacturing such a preservative.

51. Neither NCLII nor NPTI nor Paul Simmons have any right, title or interest in the product or the trademark LIQUISORB or any other wax cellulose technology (liquid preservative) product.

52. The aforesaid conduct of the defendants will likely create confusion or misunderstanding on the part of consumers, investors, and businesses.

53. As a direct and proximate result of the confusion and misunderstanding caused by defendants' deceptive practices, the Plaintiff, as the true owner of wax cellulose technology products including the product and trademark LIQUISORB, will suffer irreparable harm in the manufacture and marketing of its technology.

54. Plaintiff has no adequate remedy at law.

#### **PRAYER FOR RELIEF**

WHEREFORE, Preservation Products, LLC prays that:

- a) The Court set this matter on an expedited discovery and trial schedule;
- b) The defendants, their respective officers, directors, agents, servants, employees, successors, assigns, parent and affiliated companies, and all those controlled by or in active concert or participation with the defendants, and each of them, be preliminarily and permanently enjoined from infringing, inducing others to infringe, and contributing to the infringement of the '249, '294, '550 and '090 patents;

- c) The defendants and each of them be ordered to pay the Plaintiff damages in an amount adequate to compensate the Plaintiff for their acts of patent infringement and unfair competition, and to account for and pay over to the Plaintiff all gains and profits derived by the defendants, and each of them from their infringing acts and unfair competition;
- d) That the defendants and each of them be ordered to pay the Plaintiff increased or treble damages due to the knowing, willful and wanton nature of the defendants' patent and trademark infringement and unfair competition, and punitive damages for the willful and wanton nature of the defendants' unlawful acts;
- e) That the defendants be ordered to pay the Plaintiff pre-judgment interest on all damages awarded;
- f) That the defendants be ordered to pay the Plaintiff's attorneys' fees and costs in connection with this action;
- g) The Court grant the Plaintiff a preliminary and permanent injunction enjoining the defendants and their respective officers, directors, agents, servants, employees, successors, assigns, parent and affiliated companies, and all those controlled by or in active concert or participation with the defendants from manufacturing, marketing or claiming to manufacture and market LIQUISORB or any other natural preservative for which the defendants do not have a patent, assignment, license or other right;
- h) Order the defendants to publish on the NCLII website and in the NCLII (NCCL) Shareholder Newsletter a correction/retraction advising consumers, investors and

others that NCLII, NPTI, and Paul Simmons do not have and have never had the right to manufacture, market or sell LIQUISORB or any other similar preservative; and

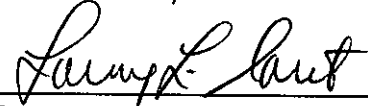
- i) For such other relief as the Court determines just and proper.

PRESERVATION PRODUCTS, LLC

By

  
Robert M. Winter

By

  
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Attorneys for Plaintiff, Preservation Products, LLC



US005840249A

**United States Patent** [19]  
**Bendiner**

[11] **Patent Number:** **5,840,249**  
[45] **Date of Patent:** **Nov. 24, 1998**

[54] **PRESERVATIVE FOR ORGANIC MATERIALS**

4,654,207 3/1987 Preston .  
5,412,090 5/1995 Bendiner ..... 536/56

[76] **Inventor:** **Bernard Bendiner, 326 Woodsedge, Suite B, Michigan City, Ind. 46360**

**FOREIGN PATENT DOCUMENTS**

131681 5/1946 Australia .  
940250 10/1963 United Kingdom .

[21] **Appl. No.:** **807,426**

[22] **Filed:** **Feb. 28, 1997**

[51] **Int. Cl.<sup>6</sup>** ..... **A61K 7/00; D21C 5/02; D21C 9/00; B01J 19/00**

[52] **U.S. Cl.** ..... **422/28; 424/401; 424/405; 162/5; 162/9; 422/40**

[58] **Field of Search** ..... **162/5, 9; 422/28, 422/40; 106/15.05; 424/405, 401**

[56] **References Cited**

**U.S. PATENT DOCUMENTS**

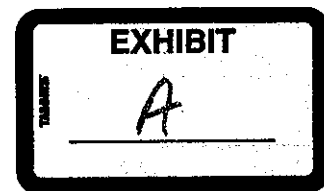
2,703,754 3/1955 Myers .  
3,248,277 4/1966 Gartner .  
3,808,089 4/1974 Von Koeppen .  
3,822,178 7/1974 Von Koeppen et al. .  
4,202,878 5/1980 Ritze .  
4,570,573 2/1986 Lohman .

*Primary Examiner*—Thurman K. Page  
*Assistant Examiner*—S. Howard  
*Attorney, Agent, or Firm*—Brinks, Hofer, Gilson & Lione

[57] **ABSTRACT**

A method for improving products such as toothpaste, shampoo, soap, detergent and lotions or creams and such improved products. The products are improved by adding a hydrous cellulose pulp that has an unlimited shelf life to the product. The hydrous cellulose pulp is resistant to decomposition and can be produced either by recycling waxed paper or through a process that begins with virgin vegetable constituents and wax. During the defibering process an emulsifier is added to the slurry and its temperature is elevated to 150°–190° Fahrenheit.

**37 Claims, No Drawings**



5,840,249

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## PRESERVATIVE FOR ORGANIC MATERIALS

### BACKGROUND OF THE INVENTION

This invention relates to a non-toxic preservative and a process for forming the non-toxic preservative. The non-toxic preservative can be added to the slurry during the paper pulping process or sprayed on the wet lap pulp in paper making process employing wet lap pulp. The preservative of this invention can also be applied to chip piles and to newly cut lumber. Another use for the preservative of this invention is to apply it to trees and bushes. Still another use is to use it in water based products such as paints and varnishes.

In the paper making industry huge wood chip piles are produced for future use in the paper making process. The wood chip piles are often stored for long periods and deterioration. Sugar present in the wood fiber is attacked by bacteria which lowers the pH, when the pH is low the wood becomes vulnerable to attack by fungi. There is great amounts of heat produced in this process and chip piles have been known to ignite as a result. In the usual paper making process biocides are added to the slurry in the pulper. The biocides slow the deterioration of the hydrous cellulose pulp material but does not stop it. In some paper making process the slurry is poured from the pulper and spread into sheets. Wet lap pulp is the term used to identify these sheets of hydrous cellulose pulp material as it emerges from a paper making pulper during the paper making process. Wet lap pulp has a very short shelf life and thus its usefulness in the non paper industry is limited. The biocides that are added to the slurry in the pulper or sprayed on the wet lap pulp are poisonous and thus the hydrous cellulose pulp material can not be used in many consumer products especially products for human consumption. Not only are the biocides not effective they are poisonous and thus present a hazard.

Most paper is made from plant fiber, most often wood, in a process that separates the cellulose from the other plant fiber material. Cellulose, the major constituent of plant fibers, is a carbohydrate. Carbohydrates are convertible into glucose by hydrolysis, a chemical process of decomposition. Under appropriate conditions the bacteria present in the paper making process contributes to and hastens decomposition. As a result, when cellulose pulp material is maintained in a hydrous state it has a very short shelf life.

In the paper making process, water is driven from the cellulose pulp and the remaining fiber is dried in a continuous operation. After the water has been removed, decomposition of the cellulose pulp ceases. However, if the process is suspended with the cellulose pulp in the hydrous state, for example over 90% water, the pulp has a very short shelf life. This short shelf life has been a major obstacle to the development of non-paper industry uses for hydrous cellulose pulp. Generally speaking, hydrous cellulose pulp is vulnerable to decomposition regardless of whether the pulp is derived from virgin vegetable constituents or from paper in a recycling operation.

Toxic biocides can be added to the process during the pulping stage which will inhibit decomposition but not stop it. The introduction of toxic biocides necessitates the addition of safety measures to protect the workers involved in the paper making process and also leaves a toxic residue in the wet lap pulp that may renders it unacceptable for use in consumer products especially consumer products that are consumed.

Furthermore, it is dangerous to spray toxic biocides on wet lap pulp after it has emerged from the pulper because the

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sprayed toxic biocides escape into and pollute the atmosphere thus creating a hazardous condition. Thus, the toxic biocides must be introduced into the paper making process during the pulping stage rather than being sprayed on the wet lap pulp after it has emerged from the pulping stage. As a result after a batch of wet lap pulp is produced that includes toxic biocides the pulping system must be cleaned to eliminate all biocides residues before a batch of wet lap pulp can be produced that is completely free of toxic biocides.

Corn starch is a very useful product when added to water for applying to the skin. However it must be used very soon after it is mixed with water or it deteriorates. This short useful life is a serious drawback to the use of corn starch.

Waxed paper is customarily manufacture by forming the paper sheet first then treating the sheet with an application of wax coating, either in dry or liquid form. For example, molten paraffin wax is easily applied by continuously passing a paper sheet through a molten bath of wax, removing the excess and then chilling. Such waxed papers have excellent resistance to water vapor, are free from odor, taste and toxicity and are low in cost.

At one time waste waxed paper presented problems in the paper recycling industry. When waste wax paper was recycled waxy spots would appear on the resulting recycled paper and a wax coating would collect on the equipment thus fouling the recycling process. Consequently, the resulting recycled paper was considered inferior and it was often necessary to stop the process so that the equipment could be adequately cleaned.

The problem, with recycling waste waxed paper, was solved however by adding a water dispersible non-ionic emulsifiers to the pulper during the repulping phase of the recycling process. The mixture containing the emulsifier is mechanically agitated at a temperature sufficiently high to melt the wax, for example from approximately 150° to 190° Fahrenheit. This process produced an emulsified wax-fiber slurry having a solids consistency of from approximately 4% to 6% by weight. The hydrous cellulose pulp produced in this process for recycling waste waxed paper has the property of an unlimited shelf life. U.S. Pat. Nos. 3,808,089 and 3,822,178, the disclosures of which are incorporated herein by reference, fully discloses the above described process.

Various non-paper industry uses have been discovered for this hydrous cellulose pulp having an unlimited shelf life. For example, as a dispersed ingredient in toothpaste, shampoo, soap, detergent, lotions and cream products. Other non-paper industry uses that were discovered for this product were its use as artificial snow and mulch. The discovery of these non-paper industry uses of hydrous cellulose pulp having an unlimited shelf life is the subject matters of U.S. Pat. No. 5,412,090 that issued on May 2, 1995. U.S. Pat. No. 5,412,090 is hereby incorporated by reference as a part of this application. The hydrous cellulose pulp having an unlimited shelf life produced in accordance with the disclosure of U.S. Pat. No. 5,412,090 has a fiber content of about 4-6% by weight. Although this fiber content function, for example when included in shampoo as a scrubbing agent, traces of the fiber that are large enough to be visible -to the consumer is left on the hair. This residue, although harmless, is unacceptable to some consumers. For the above reasons there is a need for a hydrous cellulose material that has an unlimited shelf life, that does not leave a visible residue of pulp that could render the product in which it is contained unacceptable as a consumer product and is non toxic. It was found that if the hydrous cellulose pulp formed in accordance with U.S. Pat. No. 5,412,090 is filtered, for example



5,840,249

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with a 2 micrometer filter the non-toxic filtrate contains about 0.67% hydrous cellulose pulp, in the form of minute wax coated fiber filaments and the filtrate is completely free of microorganisms. This non-toxic filtrate has been found to be an excellent water base for consumer products. This non toxic filtrate is the subject matter of co-pending application Ser. No. 08/808,212, filed on the same day as this application. The subject matter of this co-pending application Ser. No. 08/808,212 is hereby included by reference as a part of this application.

A food grade preservative commonly identified as potassium sorbate and technically identified as 2,4 Hexadienoic Acid is used for example as a preservative for food products such as pickles. Potassium sorbate is in the form of a dry powder and can be placed in solution with a water base ingredient.

There is a need for a non toxic product and method for preventing wet lap pulp from degrading and thus prolonging its shelf life.

Furthermore, there is a need for a non toxic preservative that can be sprayed on wet lap pulp that will prevent it from degrading and not contaminate the atmosphere or the wet lap pulp for use in consumer products.

A need has also become apparent for a preservative made from natural products that will stop the decomposition of carbohydrates in wet lap pulp to thereby extend its shelf life.

#### SUMMARY OF THE INVENTION

It is an object of this invention to utilize hydrous cellulose pulp having an unlimited shelf life in a process that will produce a natural ingredient preservative that is non toxic that can be added to the slurry in the pulper or sprayed on wet lap pulp to extend its shelf life and enable it to be used in consumer products.

It is another object of this invention to utilize the filtrate of hydrous cellulose pulp having an unlimited shelf life as the water base to which potassium sorbate is added to provide a preservative that can be added to the slurry in the pulper or sprayed on wet lap pulp as a replacement for the poisonous biocides.

It is a further object of this invention to provide a non-toxic preservative that can be sprayed on wet lap pulp that will extend the shelf life of the wet lap pulp sufficient to allow it to be utilized for non-paper industry processes and products.

This invention consists of a process for producing a non toxic preservative that can be sprayed on wet lap pulp that will extend the shelf life of the wet lap pulp.

This invention consists of applying the preservative of this invention to the slurry in the pulper in place of the biocides.

This invention consists of applying the preservative of this invention to chip piles and to new lumber.

This invention consists of applying the preservative of this invention to leaves and bushes to prevent mold and deterioration of the plant.

This invention consists of utilizing the preservative of this invention as the base in water base products such as paints and varnishes.

This invention further consist of a process in which the filtrate of hydrous cellulose pulp that has an unlimited shelf life is used as the water base for a liquid preservative that includes potassium sorbate.

This invention further consist of the mixture of the preservative of this invention with corn starch to extend the useful life of corn starch in liquid form.

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This invention also includes a process for producing hydrous cellulose pulp in a process including wax paper and an emulsifier along with heat sufficient to melt the wax to thus provide a micro-molecular film on the fiber, filtering the resulting hydrous cellulose pulp and combining the filtrate with potassium sorbate to produce a non-toxic liquid preservative that can be sprayed on wet lap pulp and extend its shelf life.

It is a objective of this invention to produce a non toxic preservative through a process that includes the steps of repulping waxed paper in a process requiring an emulsifier and heat sufficient to melt the wax to thus provide a micro-molecular film on the fiber resulting from the repulping process, filter out the larger fiber strands, blend the filtrate such that the minute film coated particles become dispersed in the liquid and then combining the filtrate with a food quality preservative which results in a liquid preservative that can be sprayed on wet lap pulp which can then be used in processes for producing non toxic consumer products.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

During the emulsification phase, of the wax paper recycling process used in practicing this invention, substantial quantities of wax are present from the waste waxed paper. However, this wax does not contaminate or coat the equipment even when slurries containing the emulsified product are cooled. When making waxed paper, very little wax penetrates below the surface of the un-waxed sheet of paper. However, during the emulsification phase of recycling, the paper is broken down into minute fiber filaments having irregularly shaped surfaces. Each of these minute filaments has a substantial surface area. Literally millions of fiber filaments are released from a relatively small piece of wax paper. Consequently, a piece of waxed paper having a waxed surface of 100 square inches, for example, releases fiber filaments into the emulsified slurry that have a surface area that may be as much as 1,000,000 times the original 100 square inches, or 10,000,000 square inches. The wax from the surface of the waxed paper, is melted during the emulsification phase and forms a very thin micro-molecular film on the fiber filaments. In addition to the minute fiber filaments there are numerous microorganisms from the water and other ingredients of the recycling process. The microorganisms would in the usual paper making process cause decommission of the process ingredients. However, in the process of this invention these microorganisms becomes coated with a very thin layer of wax which prevents them from causing decommission of other ingredients found in the process. This hydrous cellular pulp is 95% water, 4.67% fiber and 0.32% wax. The hydrous cellular pulp is then filtered through a 2 micrometer (0.000002 meters) filter and the resulting filtrate is then used as the water base in products such as toothpaste, shampoo, soap, detergent, lotions and cream products.

This filtrate is also used as an ingredient of a liquid preservative of this invention. The liquid preservative of this invention can be sprayed on sheets of newly produced wet lap pulp after it has been spread into sheets and or baled. This filtrate is 99% water, 0.68 fiber and 0.32% wax.

The filtrate is free of microorganisms such as bacteria and fungi, possesses an unlimited shelf life, and may be produced either by recycling waste waxed paper, new waxed paper or by processing virgin vegetable constituents in the presence of wax during the emulsification phase of the defibering process.

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A protective barrier is also believed to form around the molecular structure of the water. The filtrate, which is 99% water, and contains minute portions of fiber coated with a thin micro-molecular layer of wax derived from this process is non toxic and has an unlimited shelf life and thus can be utilized as the water base for products and provide the product with an unlimited shelf life.

In accordance with this invention, an example of the starting waxed paper that can be used is the type used in bakeries and deli-contestants to wrap food products. Waxed paper of this type is coated with a food grade paraffin wax, designated as a dry wax. Waste waxed paper can be used in the preferred embodiment and is obtained directly from the paper producing facilities. For example, trimmings from a trimming machine or wax paper that did not meet required test standards may be used. Such waxed paper is free of printing and thus is clean. The waxed paper is added to a pulper. A pulper is basically a vat for receiving a material that can be agitated by mechanical means and includes means to control the temperature. The process of pulping is essentially one of separating cells from intercellular material. It should be understood that any equipment such as a conventional high speed pulper may be used. The temperature of the wax-containing fiber slurry is raised to a temperature above the melting point of the wax and beating is continued until the wax and fiber are released into the aqueous solution. The resulting water-fiber slurry can then be subjected to a washing process to remove any impurities. Newly manufactured wax paper does not need this washing process.

The process of the present invention encompasses the use of 100% waxed paper stock having a wax content of up to 30% by weight. However, non-waxed waste paper, in modest proportions can be used without affecting the outcome. Non waxed fiber products can be used as a starting product and a paraffin wax in the correct ratio to fiber added. The use of waxed paper as a starting point has the advantage that it contains the proper ratio of fiber to wax and it is available at economical rates.

A water soluble non-ionic emulsifier is added to the pulper during the pulping phase of the paper making or recycling process. The water soluble non-ionic emulsifier being from the group consisting of: polyethylene glycol ethers of hydrophobic alcohols; alkylphenoxy polyethoxyethanols; fatty acid amides and mixtures thereof. The water soluble non-ionic emulsifier must also meet specific emulsion stability standards. The preferred water soluble non-ionic emulsifiers include: ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1; ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole of ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive; ethoxylated alkyl phenols in which the alkyl substituent is linear; and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

After the process for producing the hydrous cellulose pulp has been completed, it is filtered through a very fine filter, for example a 2 micrometer (0.000002 meters) filter to remove the larger portions of hydrous cellulose pulp, leaving a filtrate that is free of microorganism and includes only minute fiber portions that are coated with a very thin layer of wax. Although a 2 micrometer filter is used in the preferred embodiment it should be understood that a very

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fine filter is required but it need not be precisely 2 micrometers. The filtrate has a very low viscosity and can be readily sprayed through conventional nozzles.

Potassium sorbate, in powder form, is then mixed with the filtrate, at a ratio in the range of 0.1%-5% by weight, and citric acid is added until the pH is 6.5 or below. The term pH indicates the hydrogen ion concentration of a solution, which is a measure of the solution's acidity. The pH of pure water is 7.0. If acid is added to pure water, an excess of H<sub>3</sub>O<sup>+</sup> ions is formed and the acid solution has a pH ranging from 6 for a weak acid to 1 for a strong acid. Inversely, a basic solution has a low concentration of H<sub>3</sub>O<sup>+</sup> ions and an excess of OH<sup>-</sup> ions, and the pH ranges from 8 for a weak base to 14 for a strong base. Thus, the resulting preservative is a weak acid. The mixture is blended to suspend the potassium sorbate evenly throughout the filtrate.

The preservative is then sprayed on the layered or baled wet lap pulp. The wet lap pulp can then be stored and or shipped in, for example plastic bags, to processing facilities where it can undergo further processing. The wet lap pulp can be maintained in its moistened state and it will not decompose or decay. The preservative can be sprayed on the wet lap pulp and even though some preservative will escape into the atmosphere this does not pose a health hazard to the workers applying the spray or the area where the spraying is done.

Wax from the original waste waxed paper is present on minute portions of fiber that are dispersed in the preservative. The preservative coats the fibers contained in the wet lap pulp and prevents the wet lap pulp from decaying. The food grade preservative Potassium sorbate functions as a non-toxic ingredient that is carried by the slightly acid water base that alone has an unlimited shelf life. This unique combination of ingredients results in a preservative that can be easily sprayed on an organic product that is to be stored or preserved for use in the non-paper industry. Although wax is present on the minute fibers' filaments, in a very thin coating, this wax does not hinder the use of the wet lap pulp in any manner.

While the invention has heretofore been described in detail with particular reference to specific products, it is to be understood that variation, modifications and the use of equivalents can be effected without departing from the scope of this invention. It is, therefore, intended that such changes and modifications be covered by the following claims.

I claim:

1. A process for producing a preservative for applying to organic matter that will prevent the decomposition of the organic matter and enable it to be used for industrial purposes, comprising the steps of:

producing a decomposition resistant hydrous cellulose pulp, the individual fibers of which are coated with a thin wax film;

(a) filtering the decomposition resistant hydrous cellulose pulp through a very fine filter;

(b) adding potassium sorbate to the filtrate at a ratio of one part of filtrate to potassium sorbate that is in the range of 0.1% to 5% by weight;

(c) adding citric acid to the mixture in amounts to reduce the pH to 6.5 or lower.

2. The process as set forth in claim 1 wherein the organic matter being preserved is the slurry contained in the pulper during the paper making process.

3. The process as set forth in claim 1 wherein the organic matter being preserved is wet lap pulp.

4. The process as set forth in claim 1 wherein the organic matter being preserved is wood chip piles.

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5. The process as set forth in claim 1 wherein the organic matter being preserved is cut lumber.

6. The process as set forth in claim 1 wherein the organic matter being preserved is corn starch.

7. The process as set forth in claim 1 wherein the organic matter being preserved is the leaves of living plants.

8. The process as set forth in claim 1 wherein the filter through which the decomposition resistant hydrous cellulose pulp passes has opening of about 2 micrometers.

9. The process as set forth in claim 2 wherein the filter through which the decomposition resistant hydrous cellulose pulp passes has opening of about 2 micrometers.

10. The process as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

11. The process as set forth in claim 2 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

12. The process as set forth in claim 3 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

13. The process as set forth in claim 4 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

14. The process as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

15. The process as set forth in claim 2 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

16. The process as set forth in claim 3 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

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ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

17. The process as set forth in claim 4 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

18. The process as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

19. The process as set forth in claim 2 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

20. The process as set forth in claim 3 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

21. The process as set forth in claim 4 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

22. A preservative for organic matter comprising: a filtrate of a decomposition resistant hydrous

cellulose pulp material;

potassium sorbate, in the range of 0.1%-5% by weight of the filtrate; and

citric acid sufficient to lower the pH to 6.5 or lower.

23. The preservative as set forth in claim 22 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

24. The preservative as set forth in claim 22 wherein the filter through which the decomposition resistant hydrous cellulose pulp passes has opening of about 2 micrometers.

25. The preservative as set forth in claim 23 wherein the filter through which the decomposition resistant hydrous cellulose pulp passes has opening of about 2 micrometers.

26. The preservative as set forth in claim 22 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15

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carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

27. The preservative as set forth in claim 23 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

28. The preservative as set forth in claim 24 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

29. The preservative as set forth in claim 25 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

30. The preservative as set forth in claim 22 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

31. The preservative as set forth in claim 23 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

32. The preservative as set forth in claim 24 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

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ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

33. The preservative as set forth in claim 25 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

34. The preservative as set forth in claim 22 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

35. The preservative as set forth in claim 23 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

36. The preservative as set forth in claim 24 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

37. The preservative as set forth in claim 31 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

\* \* \* \* \*

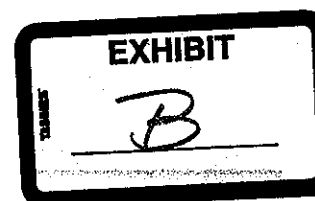
**ASSIGNMENT**

WHEREAS, BERNARD BENDINER, hereinafter called the "Assignor", has made the invention described in the United States patent entitled PRESERVATIVE FOR ORGANIC MATERIALS, for a full description of which reference is here made to Letters Patent of the United States No. 5,840,249 issued on November 24, 1998;

WHEREAS, PRESERVATION PRODUCTS, LLC, a corporation organized and existing under the laws of the State of Indiana, having a place of business at 326 Woodsedge, Suite B, Michigan City, Indiana 46360, hereinafter called the "Assignee", desires to acquire the entire right, title and interest in and to the invention and the Letters Patent identified above;

NOW, THEREFORE, in consideration of the sum of One Dollar (\$1.00), and other valuable and legally sufficient consideration, the receipt of which by the Assignor from the Assignee is hereby acknowledged, the Assignor has sold, assigned and transferred, and by these presents does sell, assign and transfer to the Assignee, the entire right, title and interest for the United States in and to the invention and the Letters Patent identified above, and all patents that may issue for said invention in the United States; together with the entire right, title and interest in and to said invention and all Letters Patents therefor in all countries foreign to the United States, including the full right to claim for any such application all benefits and priority rights under any applicable convention; together with the entire right, title and interest in and to all continuations, divisions, renewals and extensions of any of the Letters Patents defined above; to have and to hold for the sole and exclusive use and benefit of the Assignee, its successors and assigns, to the full end of the term or terms for all such patents.

The Assignor hereby covenants and agrees, for both the Assignor and the Assignor's legal representatives, that the Assignor will assist the Assignee in the making and prosecution of any other patent applications that the Assignee may elect to make covering the invention identified above; in vesting in the Assignee like exclusive title in and to all such other patent applications and patents; and in the prosecution of any interference which may



will execute and deliver to Assignee any and all additional papers which may be requested by the Assignee to carry out the terms of this Assignment.

The Commissioner of Patents and Trademarks is hereby authorized and requested to issue patents to the Assignee in accordance with the terms of this Assignment.

IN TESTIMONY WHEREOF, the Assignor has executed this agreement.

DATED:

April 7, 1999

  
BERNARD BENDINER

STATE OF ILLINOIS )  
 ) ss.  
COUNTY OF COOK)

I, Arlette Porter-Sherley, a Notary Public in and for the County and State aforesaid, do hereby certify that BERNARD BENDINER, personally known to me to be the same person whose name is subscribed to the foregoing instrument, appeared before me this day in person and acknowledged that he signed, sealed and delivered the said instrument as his free and voluntary act for the uses and purposes therein set forth.

IN WITNESS WHEREOF, I have hereunto set my hand and Notarial Seal, this 29<sup>th</sup> day of April, 1999.



Arlette Porter-Sherley  
Notary Public

My Commission Expires: June 25, 2001



US006103294A

**United States Patent** [19]  
**Bendiner**

[11] **Patent Number:** **6,103,294**  
[45] **Date of Patent:** **Aug. 15, 2000**

- [54] **PRESERVATIVE FOR DIGESTIBLE FOOD AND BEVERAGE PRODUCTS**
- [75] **Inventor:** Bernard Bendiner, Michigan City, Ind.
- [73] **Assignee:** Preservation Products, Inc., Michigan City, Ind.
- [21] **Appl. No.:** 09/099,991
- [22] **Filed:** Jun. 19, 1998

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*Primary Examiner*—Anthony J. Weier  
*Attorney, Agent, or Firm*—Brinks Hofer Gilson & Lione

**Related U.S. Application Data**

- [63] Continuation-in-part of application No. 08/807,426, Feb. 28, 1997, Pat. No. 5,840,249.
- [51] **Int. Cl.<sup>7</sup>** ..... H23L 1/222; A21D 13/00; A23K 1/00
- [52] **U.S. Cl.** ..... 426/654; 426/138; 426/615
- [58] **Field of Search** ..... 426/654, 138, 426/615

[57] **ABSTRACT**

A process for producing cellulose pulp and a filtrate of cellulose pulp that functions as a preservative for digestible food products that are intended for human and animal consumption. In the emulsification process wax paper, water, preservative potassium sorbate and the surfactant hydroxylated lecithin are heated and blended. The cellulose pulp is filtered through a filter having openings of about 2 micrometers. The cellulose pulp acts as a preservative when used with food products such as dietary fiber, a caking agent used in the dairy industry to prevent caking and clumping of graded cheese, dry seasoning and spiced soups. The cellulose pulp can also be used to improve the flowability of products which enhances their performance in packaging. The filtrate can be used as a water base for food products and acts to preserve the food product. Also fresh fruits and vegetables can be washed with the filtrate which increases the time that they can be stored without refrigeration.

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**11 Claims, No Drawings**





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## PRESERVATIVE FOR DIGESTIBLE FOOD AND BEVERAGE PRODUCTS

This application is a continuation-in-part of U.S. application Ser. No. 08/807,426, filed Feb. 28, 1997, and now U.S. Pat. No. 5,840,249, issued Nov. 24, 1998.

### BACKGROUND OF THE INVENTION

This invention relates to a preservative for digestible food products that is made from substances that are currently used in food products and generally recognized as safe. The base ingredient of this preservative is cellulose which is an indigestible carbohydrate composed of carbon, hydrogen and oxygen. The chemical terminology for this natural polymer is beta-1,4-glucan. Due to the atomic arrangements at its glycosidic bonds, the bonds linking the basic units, cellulose is insoluble in water. For all practical purposes, cellulose is considered non-caloric and is considered a GRAS (generally recognized as safe) substance by the FDA. Cellulose is the principal structural component of plants and is the most abundant source of complex carbohydrates in the world. To obtain pure cellulose, this component is progressively extracted and purified from plants. During the entire extraction process, the cellulose is not dissolved. Therefore, it exists in a naturally fibrous form and exhibits characteristics common to all fibers. The length of cellulose fibers is dependent upon the extraction process, while the typical width (diameter) of cellulose fiber is approximately 15-25 microns. Powdered cellulose is currently used in the food industry as a high fiber source and/or a non-caloric bulking agent.

Most paper is made from plant fiber, most often wood, in a process that separates the cellulose from the other plant fiber material. Cellulose is the major constituent of plant fibers. Carbohydrates, including cellulose are convertible into glucose by hydrolysis, a chemical process of decomposition. Under appropriate conditions the bacteria present in the paper making process contribute to and hasten decomposition. As a result, when cellulose pulp material is maintained in a hydrous state it has a very short shelf life.

In the paper making industry biocides are added to the slurry in the pulper. A pulper is basically a vat for receiving a material that can be agitated by mechanical means and includes means for controlling the temperature. The biocides slow the deterioration of the hydrous cellulose pulp material but do not stop it. The biocides that are added to the slurry in the pulper are poisonous and thus the hydrous cellulose pulp material can not be used in many consumer products especially products intended for human consumption. Thus, not only are the biocides not effective, they are poisonous and therefore present a hazard. In the paper making process, the water is driven from the cellulose pulp and the remaining fiber is dried in a continuous operation. After the water has been removed, decomposition of the cellulose pulp ceases. However, if the process is suspended with the cellulose pulp in the hydrous state, for example over 90% water, the pulp has a very short shelf life. This short shelf life has been a major obstacle to the development of non-paper industry uses for hydrous cellulose pulp. Generally speaking, hydrous cellulose pulp is vulnerable to decomposition regardless of whether the pulp is derived from virgin vegetable constituents or from paper in a recycling operation.

Waxed paper is customarily manufactured by forming the paper sheet first then treating the sheet with an application of wax coating, either in dry or liquid form. For example, molten paraffin wax is easily applied by continuously pass-

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ing a paper sheet through a molten bath of wax, removing the excess and then chilling. Such waxed papers have excellent resistance to water vapor, are free from odor, taste and toxicity and are low in cost.

At one time, waste or new waxed paper presented problems in the paper recycling industry. When waste or new wax paper was recycled waxy spots would appear on the resulting recycled paper and a wax coating would collect on the equipment thus fouling the recycling process. Consequently, the resulting recycled paper was considered inferior and it was often necessary to stop the process so that the equipment could be adequately cleaned.

This problem with recycling waste or new waxed paper was solved, however, by adding water dispersible non-ionic emulsifiers to the pulper during the repulping phase of the recycling process. The mixture containing the emulsifier is mechanically agitated at a temperature sufficiently high to melt the wax, for example from approximately 135° to 190° Fahrenheit. This process produced an emulsified wax-fiber slurry having a solids consistency of approximately 20% by weight. The hydrous cellulose pulp produced in this process for recycling waste or new waxed paper has the property of an unlimited shelf life. U.S. Pat. Nos. 3,808,089 and 3,822,178, the disclosures of which are incorporated herein by reference, fully disclose the above described process.

Various non-paper industry uses have been discovered for this hydrous cellulose pulp having an unlimited shelf life. For example, as a dispersed ingredient in toothpaste, shampoo, soap, detergent, lotions and cream products. Other non-paper industry uses that were discovered for this product were its use as artificial snow and mulch. The discovery of these non-paper industry uses of hydrous cellulose pulp having an unlimited shelf life is the subject matter of U.S. Pat. No. 5,412,090 that issued on May 2, 1995. U.S. Pat. No. 5,412,090 is hereby incorporated by reference as a part of this application. The hydrous cellulose pulp having an unlimited shelf life produced in accordance with the disclosure of U.S. Pat. No. 5,412,090 has a fiber content of about 4-6% by weight and has a fiber length of approximately 2,000 microns.

Powdered cellulose is used throughout the food industry for various functional purposes. It is the only dietary fiber used in the food industry. For example dietary fibers serve as a non caloric-bulking agent in numerous food products. Powdered cellulose is the standard caking agent used in the dairy industry to prevent caking and clumping of grated cheese, and more recently has gained popularity in dry seasoning, spiced soups and other mixtures. Powdered cellulose also improve the flowability of products, which enhances performance when packaging the product. However, as currently used in the food industry, powdered cellulose does not have a preservative attribute.

It was found that when hydrous cellulose pulp, produced in accordance with the disclosure of U.S. Pat. No. 5,412,090, is used in shampoo as a scrubbing agent, traces of fiber are left on the hair. These fibers are large enough to be visible to the consumer. This residue, although harmless, was found to be unacceptable to some consumers. It was found that if the hydrous cellulose pulp, formed in accordance with U.S. Pat. No. 5,412,090, is filtered, for example through a 2 micrometer filter, the non-toxic filtrate contains only about 0.67% hydrous cellulose pulp by weight. This hydrous cellulose is in the form of colloidal fibers and fibers that are a maximum of 10 microns in length. This filtrate retains the quality of an unlimited shelf life. However, the filtrate, like the fiber, is white in color. This was the result of some of the

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white wax remaining in the water phase of the filtrate. This process has been improved such that all of the wax is either on the fiber or emulsified. The colloidal fibers contained in the filtrate are transparent and thus are not, under any condition, visible to the naked eye.

Many consumer products are formed with a water base. The purest natural water includes microorganisms that will in time cause water base products to become rancid if preservatives are not added. Thus, water based consumer products commonly include a preservative. Although preservatives are chosen that most people can tolerate, some individuals are allergic to or have reactions to these preservatives. Also, the long range effect of these preservatives is often not known for certain.

A food grade preservative commonly identified as potassium sorbate and technically identified as 2,4 Hexadienoic Acid is used for example as a preservative for food products such as pickles. Potassium sorbate is commercially available in the form of a dry powder that can be placed in solution with a water based ingredient. When potassium sorbate is added to the above discussed hydrous cellulose pulp produced in accordance with the disclosure of U.S. Pat. No. 5,412,090 or its non-toxic filtrate an excellent water based preservative for consumer products is obtained. This combination of potassium sorbate and the non toxic filtrate is the subject matter of co-pending application Ser. No. 08/807,426, filed on Feb. 28, 1997. The subject matter of co-pending applications Ser. Nos. 08/808,212 and 08/807,426 are hereby included by reference as a part of this application.

There is a need for a powdered cellulose that acts as a preservative that can be used in the food industry.

There is also a need for a preservative that can be added to food products, intended for human consumption, that is made from natural ingredients that are safe and digestible.

Furthermore, there is a need for a water base that can be used for food products, made from natural digestible ingredients, that functions as a food preservative and is not visible to the naked eye.

Still further there is a need for a digestible food preservative that can be applied to the external surfaces of fresh fruits and vegetables that will extend the time that fresh fruits and vegetables can be stored in an un-refrigerated state.

#### SUMMARY OF THE INVENTION

It is an object of this invention to provide a process for producing a product and the product itself that can be used as a preservative for food intended for human as well as animal consumption.

It is also an object of this invention to provide a process for producing powdered cellulose that has a preservative attribute, and the product produced by this process, that can be used in the food industry.

It is another object of this invention to provide a process, as well as the product made by this process, that will serve as a natural ingredient preservative when applied to the outer surfaces of fresh fruits and vegetables and will not detract from the consumer's visual or taste appreciation of the fresh fruits and vegetables.

It is still another object of this invention to provide a water base for food products that will included a natural ingredient preservative and will not be visible to the human consumer.

It is another object of this invention to utilize cellulose fiber produced in a process using hydroxylated lecithin as a

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surfactant and potassium sorbate as a preservative as a dietary fiber, bulking agent, caking agent and to improve the flowability of products intended for human consumption.

It is another object of this invention to utilize the filtrate of hydrous cellulose pulp produced in a process using hydroxylated lecithin as a surfactant and potassium sorbate as a preservative as the water base for products intended for human consumption.

This invention consists of a process for producing a food grade preservative, that can be mixed in or applied to the surface of edible goods, that will extend the shelf life of the edible goods.

This invention further consist of a process in which the filtrate of hydrous cellulose pulp that has an unlimited shelf life is used as the water base for a liquid preservative that includes potassium sorbate and hydroxylated lecithin.

#### BRIEF DESCRIPTION OF THE DRAWINGS

This application does not include drawings.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

During the emulsification phase of the wax paper recycling process used in practicing this invention, substantial quantities of wax are present from the waste or new waxed paper. However, this wax does not contaminate or coat the equipment even when slurries containing the emulsified product are cooled. When making waxed paper, very little wax penetrates below the surface of the un-waxed sheet of paper. However, during the emulsification phase of recycling, the paper is broken down into minute fiber filaments having irregularly shaped surfaces. Each of these minute filaments has a substantial surface area. Literally millions of fiber filaments are released from a relatively small piece of wax paper. Consequently, a piece of waxed paper having a waxed surface of 100 square inches, for example, releases fiber filaments into the emulsified slurry that have a surface area that may be as much as 1,000,000 times the original 100 square inches, or 10,000,000 square inches. The wax from the surface of the waxed paper, is melted during the emulsification phase.

Applicant's preferred embodiment is a wax paper, that is used for bakery tissue that is manufactured by Burrows Co. This wax paper utilizes sheets of paper that is thinner than that normally used and the wax is applied to the paper under pressure such that the wax impregnates the paper fiber.

During the emulsification phase of the wax paper recycling process, a surfactant must be added to the pulper. Since this product is intended for human consumption, a food grade surfactant must be used. Applicant's preferred embodiment of food grade surfactant is a hydroxylated soybean lecithin that has high emulsifying and dispersing properties in aqueous systems.

Such an emulsifier is produced and sold by Lucas Meyer Inc. of Decatur, Ill. 62524 under their trademark "EMULFLUID HL 66". EMULFLUID HL 66 is a hydroxylated soybean lecithin with high emulsifying and dispersing properties in aqueous systems. EMULFLUID HL 66 is generally recognized as safe (GRAS) as a multi-purpose food additive under Title 21 CFR 172.814.

In the emulsification phase of the process there is a bonding of the water, wax, cellulose fiber, sorbate and the hydroxylated soybean lecithin into a mechanism that eliminates oxygen from being dissolved in the ingredient in both the fiber state and the filtrate state. Since no oxygen can be

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dissolved in the ingredient, microorganisms cannot live in this ingredient. The ingredient is not toxic and thus does not kill microorganisms, rather it inhibits their continued existence as a result of the lack of oxygen. As a result of this lack of oxygen the ingredient functions as an anti-microbial preventive and as an anti-oxidation compound. The anti-oxidation aspect of this ingredient can best be illustrated by placing a non-galvanized nail in the filtrate. The non-galvanized nail will not rust whereas if a non-galvanized nail is placed in any tap water, it will rust.

This hydrous cellulose pulp is 95% water, 4.67% fiber and 0.32% wax. The hydrous cellulose pulp can be filtered through a 2 micrometer (0.000002 meters) filter and the resulting filtrate is then used as the water base for the preservative for digestible food products. This filtrate is approximately 99% water, 0.68% fiber and 0.32% wax.

The filtrate is free of microorganisms such as bacteria and fungi, possesses an unlimited shelf life, and may be produced either by recycling waste or new waxed paper or by processing virgin vegetable constituents in the presence of wax during the emulsification phase of the defibering process.

It appears that an electric kinetic suspension has been created that has a barrier bonding mechanism that prevents oxygen from the air from dissolving in the water and the oxygen in the water from freeing itself. The filtrate is, as a result of this electric kinetic suspension, impervious to microbiological and corrosive attacks. The filtrate, which is 99% water, and contains minute portions of fiber coated with a thin micro-molecular layer of wax derived from this process, is non toxic and has an unlimited shelf life and thus can be utilized as the water base for products and provide the product with an unlimited shelf life.

In accordance with this invention, the starting waxed paper that can be used is the waxed tissue paper used in bakeries and delicatessens to wrap food products is preferred. However, any waxed paper that has been coated with a food grade paraffin wax, designated as a dry wax, can be used. Waste or new waxed paper can be used in the preferred embodiment and is obtained directly from the paper producing facilities. For example, trimmings from a trimming machine or wax paper that did not meet required test standards may be used. Such waxed paper is free of printing and thus is clean. The waxed paper is added to a pulper. A pulper is basically a vat for receiving a material that can be agitated by mechanical means and includes means to control the temperature. The process of pulping is essentially one of separating cells from intercellular material. It should be understood that any equipment such as a conventional high speed pulper may be used. The temperature of the wax-containing fiber slurry is raised to a temperature above the melting point of the wax and beating is continued until the wax and fiber are released into the aqueous solution. The resulting water-fiber slurry can then be subjected to a washing process to remove any impurities. Newly manufactured wax paper does not need this washing process.

The process of the present invention encompasses the use of 100% waxed paper stock having a wax content of up to 30% by weight. However, non-waxed waste paper, in modest proportions can be used without affecting the outcome. Non waxed fiber products can be used as a starting product and a paraffin wax in the correct ratio to fiber can be added. The use of waxed paper as a starting point has the advantage that it contains the proper ratio of fiber to wax and it is available at economical rates.

Cellulose fibers is white in color. The water phase of the filtrate includes emulsified wax which is transparent. Thus,

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the filtrate can be used as a water based food preservative, intended for human consumption, or to be applied to the external surfaces of fresh fruits and vegetables and appear as a clear liquid. It has been found that for food products intended for human consumption a water base that does not appear to be crystal clear is objectionable. Since this product is intended for human consumption, solving this problem is rendered difficult since only food grade ingredients can be utilized and even some food grade ingredients will introduce objectionable attributes into the product such as an unacceptable taste or medical side effects. Furthermore, any ingredient that is added to the product must be digestible.

After the process for producing the hydrous cellulose pulp has been completed, it is filtered through a very fine filter, for example a 2 micrometer (0.000002 meters) filter to remove the larger portions of hydrous cellulose pulp, leaving a filtrate that is free of microorganisms and includes only minute fiber portions. Although a 2 micrometer filter is used in the preferred embodiment it should be understood that a very fine filter is required but it need not be precisely 2 micrometers. The filtrate has a very low viscosity and can be readily sprayed through conventional nozzles.

While the invention has heretofore been described in detail with particular reference to specific products, it is to be understood that variations, modifications and the use of equivalents can be effected without departing from the scope of this invention. It is, therefore, intended that such changes and modifications be covered by the following claims.

I claim:

1. A cellulose pulp that acts as a preservative for digestible food products comprising:
  - a cellulose pulp material that was produced in an emulsification process from a mixture of wax paper, water, potassium sorbate, and a food grade surfactant hydroxylated soybean lecithin;
  - the emulsification process having functioned to bond the water, wax, cellulose fiber and the hydroxylated soybean lecithin into a mechanism that eliminates oxygen, thus resulting in a cellulose pulp that is free of oxygen.
2. A cellulose pulp that acts as a preservative for digestible food products as set forth in claim 1, wherein:
  - the amount of potassium sorbate is in the range of 0.1%–5% by weight of the cellulose pulp material.
3. A cellulose pulp that acts as a preservative for digestible food products as set forth in claim 1, wherein:
  - the amount of hydroxylated soybean lecithin is in the range of 0.01% to 0.10% by weight of the cellulose pulp material.
4. A cellulose pulp that acts as a preservative for digestible food products as set forth in claim 2 wherein the amount of hydroxylated soybean lecithin is in the range of 0.01% to 0.10% by weight of the cellulose pulp material.
5. A preservative for digestible food products comprising:
  - a filtrate of a decomposition resistant hydrous cellulose pulp material that was produced in an emulsification process from a mixture of wax paper, water, potassium sorbate, and hydroxylated soybean lecithin;
  - the emulsification process having functioned to bond the water, wax, cellulose fiber and the hydroxylated soybean lecithin into a mechanism that eliminates oxygen, thus resulting in a cellulose pulp that is free of oxygen.
6. A preservative for digestible food products as set forth in claim 5 wherein the amount of potassium sorbate is in the range of 0.1%–5% by weight of the filtrate.
7. A preservative for digestible food products as set forth in claim 5 wherein the amount of hydroxylated soybean lecithin is in the range of 0.01% to 0.10% by weight of the filtrate.

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8. A preservative for digestible food products as set forth in claim 6 wherein the amount of hydroxylated soybean lecithin is in the range of 0.01% to 0.10% by weight of the filtrate.

9. The preservative for digestible food products as set forth in claim 5 wherein:

the filtrate of a decomposition resistant hydrous cellulose pulp material has passed through a filter that has opening of about 2 micrometers.

10. The preservative for digestible food products as set forth in claim 5 wherein:

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said filtrate of a decomposition resistant hydrous cellulose pulp material is filtered from a decomposition resistant hydrous cellulose pulp material that was produced in a process that used a water soluble non-ionic emulsifier.

11. The preservative for digestible food products as set forth in claim 6 wherein:

said filtrate of a decomposition resistant hydrous cellulose pulp material is filtered from a decomposition resistant hydrous cellulose pulp material that was produced in a process that used a water soluble non-ionic emulsifier.

\* \* \* \* \*

**ASSIGNMENT**

WHEREAS, BERNARD BENDINER, hereinafter called the "Assignor", has made the invention described in the United States patent application entitled PRESERVATIVE FOR DIGESTIBLE FOOD AND BEVERAGE PRODUCTS, for a full description of which reference is here made to an application for Letters Patent of the United States filed on June 19, 1998 and assigned Application Serial No. 09/099,991;

WHEREAS, PRESERVATION PRODUCTS, LLC, a corporation organized and existing under the laws of the State of Indiana, having a place of business at 326 Woodsedge, Suite B, Michigan City, Indiana 46360, hereinafter called the "Assignee", desires to acquire the entire right, title and interest in and to the invention and the patent application identified above, and all patents which may be obtained for said invention, as set forth below;

NOW, THEREFORE, in consideration of the sum of One Dollar (\$1.00), and other valuable and legally sufficient consideration, the receipt of which by the Assignor from the Assignee is hereby acknowledged, the Assignor has sold, assigned and transferred, and by these presents does sell, assign and transfer to the Assignee, the entire right, title and interest for the United States in and to the invention and the patent application identified above, and any patents that may issue for said invention in the United States; together with the entire right, title and interest in and to said invention and all patent applications and patents therefor in all countries foreign to the United States, including the full right to claim for any such application all benefits and priority rights under any applicable convention; together with the entire right, title and interest in and to all continuations, divisions, renewals and extensions of any of the patent applications and patents defined above; to have and to hold for the sole and exclusive use and benefit of the Assignee, its successors and assigns, to the full end of the term or terms for all such patents.

The Assignor hereby covenants and agrees, for both the Assignor and the Assignor's legal representatives, that the Assignor will assist the Assignee in the prosecution of the patent application identified above; in the making and prosecution of any other patent applications that the Assignee may elect to make covering the invention identified above; in



vesting in the Assignee like exclusive title in and to all : other ent applications and patents; and in the prosecution of any interference which may arise involving said invention, or any such patent application or patent; and that the Assignor will execute and deliver to the Assignee any and all additional papers which may be requested by the Assignee to carry out the terms of this Assignment.

The Commissioner of Patents and Trademarks is hereby authorized and requested to issue patents to the Assignee in accordance with the terms of this Assignment.

IN TESTIMONY WHEREOF, the Assignor has executed this agreement.

DATED:

April 29, 1999

  
BERNARD BENDINER

STATE OF ILLINOIS) )  
 ) ss.  
COUNTY OF COOK)

I, Arlette Porter-Sherley, a Notary Public in and for the County and State aforesaid, do hereby certify that BERNARD BENDINER, personally known to me to be the same person whose name is subscribed to the foregoing instrument, appeared before me this day in person and acknowledged that he signed, sealed and delivered the said instrument as his free and voluntary act for the uses and purposes therein set forth.

IN WITNESS WHEREOF, I have hereunto set my hand and Notarial Seal, this 29<sup>th</sup> day of April, 1999.



*Arlette Porter-Sherley*  
\_\_\_\_\_  
Notary Public

My Commission Expires: June 25, 2001



US006171550B1

(12) **United States Patent**  
**Bendiner**

(10) **Patent No.: US 6,171,550 B1**  
(45) **Date of Patent: \*Jan. 9, 2001**

(54) **NON-TOXIC BASE INGREDIENT FOR CONSUMER PRODUCTS**

5,491,190 \* 2/1996 Sandvick et al. .... 536/56  
5,840,249 \* 11/1998 Bendiner ..... 422/28

(75) **Inventor: Bernard Bendiner, Michigan City, IN (US)**

**FOREIGN PATENT DOCUMENTS**

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(73) **Assignee: Preservation Products, LLC, Michigan City, IN (US)**

**OTHER PUBLICATIONS**

(\*) **Notice: Under 35 U.S.C. 154(b), the term of this patent shall be extended for 0 days.**

Sato et al, Toothpastes containing granules which change flavor and taste during tooth-brushing. CAPLUS AN 1987:561426 abs., May 28, 1997.\*

This patent is subject to a terminal disclaimer.

\* cited by examiner

(21) **Appl. No.: 08/808,212**

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(22) **Filed: Feb. 28, 1997**

(74) *Attorney, Agent, or Firm—Brinks Hofer Gilson & Lione*

(51) **Int. Cl.<sup>7</sup> ..... A61K 7/00; D21C 5/02; D21C 9/00; B01J 19/00**

(57) **ABSTRACT**

(52) **U.S. Cl. .... 422/28; 424/401; 424/405; 162/5; 162/9; 422/40**

A method for improving products such as toothpaste, shampoo, soap, detergent and lotions or creams by utilizing a water base for the product that is about 99% water, 0.68% cellulose and 0.32% emulsified food grade wax. Products produced with this water base will not decompose even though chemical preservatives is not included as an ingredient. The water base is formed by filtering hydrous cellulose pulp that is resistant to decomposition through a very fine filter. The hydrous cellulose pulp can be produced either by recycling waxed paper or thorough a process that begins with virgin vegetable constituents and wax. The filtrate, or water base can also be used in the horticultural field to prevent mold on plants and in physiology area to prevent the degradation of cells.

(58) **Field of Search ..... 424/401, 70.1, 424/405; 422/28, 40; 162/5, 9**

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**8 Claims, No Drawings**





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## NON-TOXIC BASE INGREDIENT FOR CONSUMER PRODUCTS

### BACKGROUND OF THE INVENTION

This invention relates to a non-toxic ingredient and the process for producing this ingredient that can be used as the base in consumer products. When this ingredient is used as the base it is not necessary to add preservatives to the consumer product.

Most paper is made from plant fiber, most often wood, in a process that separates the cellulose from the other plant fiber material. Cellulose, the major constituent of plant fibers, is a carbohydrate. Carbohydrates are convertible into glucose by hydrolysis, a chemical process of decomposition. Under appropriate conditions the bacteria present in the paper making process contributes to and hastens decomposition. As a result, when cellulose pulp material is maintained in a hydrous state it has a very short shelf life.

In the paper making process, water is driven from the cellulose pulp and the remaining fiber is dried in one continuous operation. After the water has been removed, decomposition of the cellulose pulp ceases. However, if the process is suspended with the cellulose pulp in the hydrous state, for example over 90% water, the pulp has a very short shelf life. This short shelf life has been a major obstacle to the development of non-paper industry uses for hydrous cellulose pulp.

Generally speaking, hydrous cellulose pulp is vulnerable to decomposition regardless of whether the pulp is derived from virgin vegetable constituents or from paper in a recycling operation.

Toxic biocides are added to the process during the pulping stage which will inhibit decomposition but not stop it. The introduction of toxic biocides necessitated the addition of safety measures to protect the workers involved in the paper making process. In some paper making processes a slurry of hydrous cellulose pulp material is spread in sheets and baled for shipping. Wet lap pulp is the term used to identify this hydrous cellulose pulp material. If biocides are sprayed on the wet lap pulp after it has emerged from the pulper, the toxic biocides escape into the atmosphere and/or flow into our streams and rivers and thus create hazardous conditions. Furthermore, toxic residues remain in the wet lap pulp that may renders it unacceptable for use in consumer products especially consumer products that are consumed. Thus, the toxic biocides must be introduced into the paper making process during the pulping stage rather than being sprayed on the wet lap pulp after it has emerged from the pulping stage. As a result after a batch of wet lap pulp is produced that includes toxic biocides the pulping system must be cleaned to eliminate all biocides residues before a batch of wet lap pulp can be produced that is completely free of toxic biocides.

Waxed paper is customarily manufacture by forming the paper sheet first then treating the sheet with an application of wax coating, either in dry or liquid form. For example, molten paraffin wax is easily applied by continuously passing a paper sheet through a molten bath of wax, removing the excess and then chilling. Such waxed papers have excellent resistance to water vapor, are free from odor, taste and toxicity and are low in cost.

At one time waste waxed paper presented problems in the paper recycling industry. When waste wax paper was recycled waxy spots would appear on the resulting recycled paper and a wax coating would collect on the equipment thus fouling the recycling process.

Consequently, the resulting recycled paper was considered inferior and it was often necessary to stop the process so that the equipment could be adequately cleaned.

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This problem, with recycling waste waxed paper, was solved however by adding a water dispersible non-ionic emulsifiers to the pulper during the repulping phase of the recycling process. The mixture containing the emulsifier is mechanically agitated at a temperature sufficiently high to melt the wax, for example from approximately 150° to 190° Fahrenheit. This process produced an emulsified wax-fiber slurry having a solids consistency of from approximately 4% to 6% by weight. The hydrous cellulose pulp produced in this process for recycling waste waxed paper has the property of an unlimited shelf life. U.S. Pat. Nos. 3,808,089 and 3,822,178, the disclosures of which are incorporated herein by reference, fully discloses the above described process.

Various non-paper industry uses have been discovered for this hydrous cellulose pulp having an unlimited shelf life. For example, as a dispersed ingredient in toothpaste, shampoo, soap, detergent, lotions and cream products. Other non-paper industry uses that were discovered for this product were its use as artificial snow and mulch. The discovery of these non-paper industry uses of hydrous cellulose pulp having an unlimited shelf life is the subject matters of U.S. Pat. No. 5,412,090 that issued on May 2, 1995. U.S. Pat. No. 5,412,090 is hereby incorporated by reference as a part of this application. The hydrous cellulose pulp having an unlimited shelf life produced in accordance with the disclosure of U.S. Pat. No. 5,412,090 has a fiber content of about 4-6% by weight. Although this fiber content function, for example when included in shampoo as a scrubbing agent, traces of the fiber that are large enough to be visible to the consumer is left on the hair. This residue, although harmless, is unacceptable to some consumers. For the above reasons there is a need for a hydrous cellulose material that has an unlimited shelf life, that does not leave a visible pulp residue that could render the product in which it is contained unacceptable as a consumer product and is non toxic.

Many consumer products are formed with a water base. The purest natural water includes microorganisms that will in time cause water base products to become rancid if preservatives are not added. Thus, water based consumer products commonly include a preservative. Although preservatives are chosen that most people can tolerate, some individuals are allergic to or have reactions to these preservatives. Also, the long range effect of these preservatives is often not know for certain.

Thus, there is a need for a non toxic product that can server as an exfoliant or emollient agent in consumer products that does not leave a visible residue.

Furthermore, there is a need for a base ingredient that can be used in consumer products that does not require a preservative to prevent the consumer product from degrading and not contaminate the hydrous cellulose material for use in consumer products.

There is also a need in the horticultural field to prevent mold on plants and in physiology area to prevent the degradation of cells.

### SUMMARY OF THE INVENTION

It is an object of this invention to utilize hydrous cellulose pulp in a process that will produce a natural base ingredient that includes colloidal cellulose particles that do not require a preservative and can be used as the water base in consumer products.

It is another object of this invention to provide a method for improving toothpaste, shampoo, soap, detergent, lotions and cream products by using a liquid base ingredient that contains about 0.68% colloidal cellulose particles that has an unlimited shelf life and that functions as a natural ingredient preservative for the other ingredients of the product, is not toxic, does not leave a visible residue, and is soft and soothing to the skin or mucous membrane.

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It is a further object of this invention to provide a process for improving toothpaste, shampoo, soap, detergent, lotions and cream products that includes the steps of repulping waxed paper in a process requiring an emulsifier and heat sufficient to melt the wax to thus provide a micro-molecular film on the fiber, filter out the fiber, using a 2 micrometer filter and then utilize the filtrate which contains minute wax coated fiber filaments as the water base ingredient in the process for producing non toxic consumer products.

When this filtrate is used for example as the water base for hair spray a molecular film of food grade wax is left on the users hair which makes the hair easier to groom.

It is a further object of this invention to provide a process for producing a water base that has the quality of opacity which can be used as the water base in products such as toothpaste, shampoo, soap, detergent, lotions and cream products. This process includes the steps of repulping waxed paper in a process requiring an emulsifier and heat sufficient to melt the wax to thus provide a micro-molecular film on the fiber resulting from the repulping process, filter out the visible fiber strands, blending the filtrate such that the minute film coated particles become dispersed in the liquid and then utilize the liquid with the dispersed film coated particles as the water base ingredient in consumer products.

It is a still further object of this invention to provide a process for improving water based products such as toothpaste, shampoo, soap, detergent, lotions and cream products by utilizing in the process for forming the product a water base ingredient that is completely free of microorganisms and includes minute particles of fiber dispersed therein that are coated with a thin wax film.

It is an object of this invention to utilize a filtrate of hydrous cellulose pulp that has an unlimited shelf life in a process that will function as a natural ingredient preservative to the other ingredients contained in the process and improve the quality of the product.

It is another object of this invention to provide a method for improving toothpaste, shampoo, soap, detergent lotions and cream products by using a liquid base that contains about 0.68% colloidal cellulose particles and about 0.32% emulsified food grade wax that function as a natural preservative to the other ingredients of the product and does not leave a visible residue.

It is a further object of this invention to provide a process for improving low viscosity products such as mouthwash or glass cleaners that include the steps of forming colloidal cellulose particles that has an unlimited shelf life from natural materials in the presence of wax in a process requiring an emulsifier and heat for increasing the temperature sufficient to melt the wax to thus provide a micro-molecular film on the fiber and then blend and filter the hydrous cellulose pulp and utilize the resulting filtrate as the water base ingredient for the low viscosity products.

Another object of this invention is to prevent mold on plants and trees by applying the filtrate as a mulch for and a water source for the plants.

It is yet another object of this invention to use the filtrate to protect healthy cells in animals and humans from attack by cells that have been degraded by cancer, aids and viruses that degrade cells.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

During the emulsification phase, of the wax paper recycling process used in practicing this invention, and more fully disclosed in the above identified U.S. Pat. No. 5,412,090, substantial quantities of wax are present from the waste waxed paper. However, this wax does not contaminate or coat the equipment even when slurries containing the emul-

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sified product are cooled. When making waxed paper, very little wax penetrates below the surface of the un-waxed sheet of paper. However, during the emulsification phase of recycling, the paper is broken down into minute fiber filaments having irregularly shaped surfaces. Each of these minute filaments has a substantial surface area. Literally millions of fiber filaments are released from a relatively small piece of wax paper. Consequently, a piece of waxed paper having a waxed surface of 100 square inches, for example, releases fiber filaments into the emulsified slurry that have a surface area that may be as much as 1,000,000 times the original 100 square inches, or 10,000,000 square inches. The wax from the surface of the waxed paper, is melted during the emulsification phase and forms a very thin micro-molecular film on the fiber filaments. In addition to the minute fiber filaments there are numerous microorganisms from the water and other ingredients of the recycling process. The microorganisms would in the usual paper making process cause decommission of the process ingredients. However, in the process of this invention these microorganisms becomes coated with a very thin layer of wax which prevents them from causing decommission of other ingredients found in the process. This hydrous cellulose pulp is 95% water, 4.67% fiber and 0.32% wax. The hydrous cellulose pulp is then filtered through a very fine filter, for example a 2 micrometer (0.000002 meters) filter and the resulting filtrate is then used as the water base in products such as toothpaste, shampoo, soap, detergent, lotions and cream products. This filtrate is 99% water, 0.68 colloidal cellulose particles and 0.32% emulsified food grade wax. A colloidal dispersion is thus formed having colloidal cellulose particles that are smaller than 35 microns. The colloidal cellulose particles are not visible to the naked eye. The filtrate is an emollient which is soothing to the skin or mucous membrane.

This hydrous cellulose pulp and the filtrate thereof is free of microorganisms such as bacteria and fungi, possesses an unlimited shelf life, and may be produced either by recycling waste waxed paper, new waxed paper or by processing virgin vegetable constituents in the presence of wax during the emulsification phase of the defibering process.

A protective barrier is also believed to form around the molecular structure of the water. The filtrate contains minute portions of fiber coated with a thin micro-molecular layer of wax derived from this process is non toxic and has an unlimited shelf life and thus can be utilized as the water base ingredient for consumer products and provide the product with an unlimited shelf life.

The filtrate functions as a water base that is free of biological microorganisms and includes minute portions of fiber that has been coated with a thin micromolecular layer of wax. The minute portions of wax coated fiber function as scrubbing agents in shampoos, soaps and detergents. The resulting product has an unlimited shelf life and is non toxic.

In accordance with this invention, an example of the type of waxed paper that can be used, in the practice of this invention is the type used in bakeries and delicatessens to wrap food products. Waxed paper of this type is coated with a food grade paraffin wax, designated as a dry wax. Waste waxed paper can be used in the preferred embodiment that is obtained directly from the paper producing facilities. For example, trimmings from a trimming machine or wax paper that did not meet required test standards may be used. Such waxed paper is free of printing and thus is clean. The waxed paper is added to a pulper. A pulper is basically a vat for receiving a material that can be agitated by mechanical means and includes means to control the temperature. The process of pulping is essentially one of separating cells from intercellular material. It should be understood that any equipment such as a conventional high speed pulper may be

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used. The temperature of the wax-containing fiber slurry is raised to a temperature above the melting point of the wax. The slurry is beat until the wax and fiber are released into the aqueous solution. The resulting water-fiber slurry can then be subjected to a washing process to remove any impurities. Newly manufactured waxed paper does not need this washing process.

The process of the present invention encompasses the use of 100% waxed paper stock having a wax content of up to 30% by weight. However, non-waxed waste paper, in modest proportions can be used without affecting the outcome. Non waxed fiber products can be used as a starting product and a paraffin wax in the correct ratio to fiber added. The use of waxed paper as a starting point has the advantage that it contains the proper ratio of fiber to wax and it is available at economical rates.

The water soluble non-ionic emulsifier that is added to the slurry being from the group consisting of: polyethylene glycol ethers of hydrophobic alcohols; alkylphenoxy polyethoxyethanols; fatty acid amides and mixtures thereof. The water soluble non-ionic emulsifier must also meet specific emulsion stability standards. The preferred water soluble non-ionic emulsifiers include: ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1; ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive; ethoxylated alkyl phenols in which the alkyl substituent is linear; and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

After the process for producing the hydrous cellulose pulp has been completed, it is filtered through a 2 micrometer filter to remove the larger portions of hydrous cellulose pulp, leaving a filtrate comprised of colloidal cellulose particles that are smaller than 35 microns. This filtrate is free of microorganism and the colloidal cellulose particles are coated with a very thin layer of wax. This filtrate is mixed to suspend the coated fiber in the liquid. This filtrate can then be utilized as the water base ingredient in products such as toothpaste, shampoo, soap, detergent, lotions or creams. Wax from the original waste waxed paper is present on the minute portions of fiber that are dispersed in the final product. However, such wax is present on the minute fibers filaments, in a very thin coating. Although a 2 micrometer filter is used in the preferred embodiment it should be understood that although a very fine filter is necessary it can vary within limits of 2 micrometers.

The filtrate can also be used in the horticulture area. The filtrate is used to mulch or water plants and the wax coated colloidal cellulose particles are synthesized by the roots and work their way up to the leaves. It has been demonstrated that mold that exist on leaves prior to the application of the filtrate does not permeate to the healthy portions of the leaf. The wax coated colloidal cellulose particles forms a molecular film around the plant cells and protects them from attack by the mold.

It is felt that, consistent with known similar situations in the biological field, the results found in the biological plant world will be duplicated in the biological animal world. It is felt, for example, that the wax coated colloidal cellulose particles will be synthesized in animals including humans and a film will be formed around healthy cells that will protect them from attack by cells that have been degraded. Thus, this invention contemplates a process for retarding cellular deterioration in animals, including humans, in which the cells are degrading as a result of cancer, aids or viruses that create cellular degradation such as hepatitis.

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While the invention has heretofore been described in detail with particular reference to specific products, it is to be understood that variation, modifications and the use of equivalents can be effected without departing from the scope of this invention. It is, therefore, intended that such changes and modifications be covered by the following claims.

I claim:

1. A process for improving toothpaste, shampoo, soap, detergent and lotions or creams comprising the steps of:

(a) producing a decomposition resistant hydrous cellulose pulp, the individual fibers of which are coated with a thin wax film; and

(b) filtering the hydrous cellulose pulp through a fine filter; and

(c) utilizing the filtrate as the water base in toothpaste, shampoo, soap, detergent and lotions or creams.

2. The invention as set forth in claim 1 wherein said fine filter has openings that are about 2 micrometers in size.

3. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

4. The invention as set forth in claim 2 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

5. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

6. The invention as set forth in claim 2 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

7. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

8. The invention as set forth in claim 2 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

\* \* \* \* \*

**ASSIGNMENT**

WHEREAS, BERNARD BENDINER, hereinafter called the "Assignor", has made the invention described in the United States patent application entitled NON-TOXIC BASE INGREDIENT FOR CONSUMER PRODUCTS, for a full description of which reference is here made to an application for Letters Patent of the United States filed on February 28, 1997 and assigned Application Serial No. 08/808,212;

WHEREAS, PRESERVATION PRODUCTS, LLC, a corporation organized and existing under the laws of the State of Indiana, having a place of business at 326 Woodsedge, Suite B, Michigan City, Indiana 46360, hereinafter called the "Assignee", desires to acquire the entire right, title and interest in and to the invention and the patent application identified above, and all patents which may be obtained for said invention, as set forth below;

NOW, THEREFORE, in consideration of the sum of One Dollar (\$1.00), and other valuable and legally sufficient consideration, the receipt of which by the Assignor from the Assignee is hereby acknowledged, the Assignor has sold, assigned and transferred, and by these presents does sell, assign and transfer to the Assignee, the entire right, title and interest for the United States in and to the invention and the patent application identified above, and any patents that may issue for said invention in the United States; together with the entire right, title and interest in and to said invention and all patent applications and patents therefor in all countries foreign to the United States, including the full right to claim for any such application all benefits and priority rights under any applicable convention; together with the entire right, title and interest in and to all continuations, divisions, renewals and extensions of any of the patent applications and patents defined above; to have and to hold for the sole and exclusive use and benefit of the Assignee, its successors and assigns, to the full end of the term or terms for all such patents.

The Assignor hereby covenants and agrees, for both the Assignor and the Assignor's legal representatives, that the Assignor will assist the Assignee in the prosecution of the patent application identified above; in the making and prosecution of any other patent applications that the Assignee may elect to make covering the invention identified above; in

EXHIBIT

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vesting in the Assignee like exclusive title in and to all such other patent applications and patents; and in the prosecution of any interference which may arise involving said invention, or any such patent application or patent; and that the Assignor will execute and deliver to the Assignee any and all additional papers which may be requested by the Assignee to carry out the terms of this Assignment.

The Commissioner of Patents and Trademarks is hereby authorized and requested to issue patents to the Assignee in accordance with the terms of this Assignment.

IN TESTIMONY WHEREOF, the Assignor has executed this agreement.

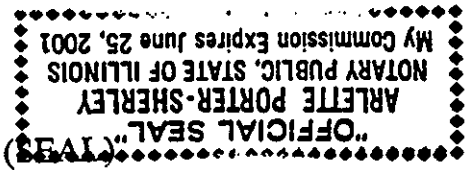
DATED: April 7, 1999

  
BERNARD BENDINER

STATE OF ILLINOIS )  
 ) ss.  
COUNTY OF COOK)

I, Arlette Porter-Sherley, a Notary Public in and for the County and State aforesaid, do hereby certify that BERNARD BENDINER, personally known to me to be the same person whose name is subscribed to the foregoing instrument, appeared before me this day in person and acknowledged that he signed, sealed and delivered the said instrument as his free and voluntary act for the uses and purposes therein set forth.

IN WITNESS WHEREOF, I have hereunto set my hand and Notarial Seal, this 29<sup>th</sup> day of April, 1999.



Arlette Porter-Sherley  
Notary Public

My Commission Expires: June 25, 2001





US005412090A

# United States Patent [19]

[11] Patent Number: **5,412,090**

**Bendiner**

[45] Date of Patent: **May 2, 1995**

[54] **HYDROUS CELLULOSE PULP FOR NON PAPER PRODUCTS**

4,570,573 2/1986 Lohman ..... 119/1  
4,654,207 3/1987 Preston ..... 424/70

[76] Inventor: **Bernard Bendiner, 8815 W. Golf Rd., Suite 12D, Niles, Ill. 60714**

### FOREIGN PATENT DOCUMENTS

[21] Appl. No.: **190,301**

131681 5/1946 Australia .  
0174825 3/1986 European Pat. Off. .  
940250 10/1963 United Kingdom .

[22] Filed: **Feb. 2, 1994**

[51] Int. Cl.<sup>6</sup> ..... **A61K 7/06; A61K 7/16; A61K 7/50**

*Primary Examiner*—Ronald W. Griffin  
*Attorney, Agent, or Firm*—Willian Brinks Hofer Gilson & Lione

[52] U.S. Cl. .... **536/56; 162/5; 424/49; 424/70.13; 252/14; 252/32; 252/89.1; 252/108; 252/128; 252/130; 252/DIG. 13; 252/DIG. 5; 252/DIG. 10**

### [57] ABSTRACT

[58] Field of Search ..... **162/5; 536/56; 514/781, 514/846, 881; 424/49, 70; 252/128, 130, DIG. 5, DIG. 10, DIG. 13, 14, 32, 89.1, 108**

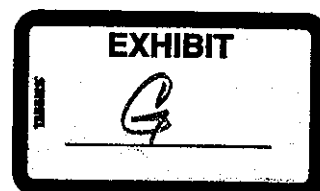
A method for improving products such as toothpaste, shampoo, soap, detergent and lotions or creams and such improved products. The products are improved by adding a hydrous cellulose pulp that has an unlimited shelf life to the product. The hydrous cellulose pulp is resistant to decomposition and can be produced either by recycling waxed paper or through a process that begins with virgin vegetable constituents and wax. During the defibering process an emulsifier is added to the slurry and its temperature is elevated to 150°-190° Fahrenheit.

### [56] References Cited

#### U.S. PATENT DOCUMENTS

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3,248,277 4/1966 Gärtner ..... 162/5  
3,808,089 4/1974 Von Koeppen et al. .... 162/5  
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**14 Claims, No Drawings**



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## HYDROUS CELLULOSE PULP FOR NON PAPER PRODUCTS

### BACKGROUND OF THE INVENTION

This invention relates to a process for producing pulp, having unique proprieties, from virgin wood constituents or from waxed paper and using the pulp as an additive for non-paper industry type products such as toothpaste, shampoo, soap, detergent and lotions or creams. The pulp also can be used for toys, ground cover and to simulate snow.

Most paper is made from plant fiber, most often wood, in a process that separates the cellulose from the other plant fiber material. Cellulose, the major constituent of plant fibers, is a carbohydrate. Carbohydrates are convertible into glucose by hydrolysis; a chemical process of decomposition. Under appropriate conditions the bacteria present in the paper making process contributes to and hastens decomposition. As a result, cellulose pulp material maintained in a hydrous state has a very short shelf life.

In the paper making process, water is driven from the cellulose pulp and the remaining fiber is dried in one continuous operation. Thus decomposition of the cellulose pulp is not a problem. However, if the process is suspended with the cellulose pulp in the hydrous state, for example over 90% water, the pulp has a very short shelf life. This short shelf life has been a major obstacle to the development of non-paper industry uses for hydrous cellulose pulp. Generally speaking, hydrous cellulose pulp is vulnerable to decomposition regardless whether the pulp is derived from virgin vegetable constituents or from a paper recycling operation.

Waxed paper is customarily manufacture by forming the paper sheet first then treating the sheet with an application of wax coating, either in dry or liquid form. For example, molten paraffin wax is easily applied by continuously passing a paper sheet through a molten bath of wax, removing the excess and then chilling. Such waxed papers have excellent resistance to water vapor, are free from odor, taste and toxicity and are low in cost.

At one time waste waxed paper presented problems in the paper recycling industry. When waste wax paper was recycled waxy spots would appear on the resulting recycled paper and a wax coating would collect on the equipment thus fouling the recycling process. Consequently, the resulting recycled paper was considered inferior and it was often necessary to stop the process so that the equipment could be adequately cleaned.

The problem, with recycling waste waxed paper, was solved however by adding a water dispersible non-ionic emulsifiers to the pulper during the repulping phase of the recycling process. The mixture containing the emulsifier is mechanically agitated at a temperature sufficiently high to melt the wax, for example from approximately 150° to 190° Fahrenheit. This process produced an emulsified wax-fiber slurry having a solids consistency of from approximately 4% to 6% by weight. U.S. Pat. Nos. 3,808,089 and 3,822,178, the disclosures of which are incorporated herein by reference, fully discloses the above described process. As is discussed in U.S. Pat. No. 3,822,178, the water soluble non-ionic emulsifiers used in the process are selected from the group consisting of polyethylene glycol ethers of hydrophobic alcohols, alkylphenoxy polyethoxyethanols, fatty acid amides and mixtures thereof and meet the

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following emulsion stability standard. Emulsifiers for practicing this invention include: the ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1; ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive; ethoxylated alkyl phenols in which the alkyl substituent is linear; and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

The hydrous cellulose pulp produced in this process for recycling waste waxed paper has the property of an unlimited shelf life.

### SUMMARY OF THE INVENTION

It is an object of this invention to utilize hydrous cellulose pulp that has an unlimited shelf life in processes and products outside of the paper industry.

It is another object of this invention to provide a method for improving toothpaste, shampoo, soap, detergent lotions and cream products by adding hydrous cellulose pulp that has an unlimited shelf life when the product is in liquid form such that the pulp can become dispersed evenly throughout the product.

It is a further object of this invention to provide a process for improving toothpaste, shampoo, soap, detergent lotions and cream products that include the steps of repulping waxed paper in a process requiring an emulsifier and heat sufficient to melt the wax to thus provide a micro-molecular film on the fiber and then dispersing the fiber in the product when it is in a liquid form.

It is a still further object of this invention to provide a hydrous cellulose pulp material that, at temperatures above freezing, simulates snow.

It is a further object of this invention to provide a mulch for plants that will maintain a high moisture level and can be dyed such that its color can be earthen or be coordinated with the plant's color.

It is a further object of this invention to improve the efficiency of toothpaste in both its ability to cleanse teeth and its ability to stimulate and condition the gums by a process that include the steps of forming hydrous cellulose pulp that has an unlimited shelf life in the presence of wax in a process requiring an emulsifier and heat; for increasing the temperature sufficient to melt the wax to thus provide a micro-molecular film on the fiber and then blend and disperse the fiber in the toothpaste.

It is a further object of this invention to provide a process for improving products such as mouthwash or glass cleaners that have a low viscosity that include the steps of forming hydrous cellulose pulp that has an unlimited shelf life from material in the presence of wax in a process requiring an emulsifier and heat for increasing the temperature sufficient to melt the wax to thus provide a micro-molecular film on the fiber and then blend and disperse the fiber such that it becomes suspended in the product.

It is a further object of this invention to provide a process for improving lotions and creams that include the steps of forming hydrous cellulose pulp that has an unlimited shelf life from material in the presence of wax



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in a process requiring an emulsifier and heat for increasing the temperature sufficient to melt the wax to thus provide a micro-molecular film on the fiber and then blend and disperse the fiber in the lotion or cream.

#### DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

During the emulsification phase, of the wax paper recycling process used in practicing this invention, substantial quantities of wax are present from the waste waxed paper. However, this wax does not contaminate or coat the equipment even when slurries containing the emulsified product are cooled. When making waxed paper, very little wax penetrates below the surface of the unwaxed sheet of paper. However, during the emulsification phase of recycling, the paper is broken down into minute fiber filaments having irregularly shaped surfaces. Each of these minute filaments has a substantial surface area. Literally millions of fiber filaments are released from a relatively small piece of wax paper. Consequently, a piece of waxed paper having a waxed surface of 100 square inches, for example, releases fiber filaments into the emulsified slurry that have a surface area that may be as much as 1,000,000 times the original 100 square inches, or 10,000,000 square inches. The wax from the surface of the waxed paper, is melted during the emulsification phase and forms a very thin micro-molecular film on the fiber filaments. As a result of this micro-molecular film, paper products made in accordance with this product are characterized by the complete absence of glossy specks even though they were formed from 100% high wax content stock. Thus, although the original wax is still present after the emulsification process, its presence is not apparent. In the past, the pulp formed in this process was used exclusively in the paper industry for making high quality recycled paper products. However a dramatic change has occurred in the cellulose pulp, it is no longer vulnerable to decomposition. The very thin micro-molecular film on the fibers produced in this process has rendered the cellulose pulp immune from the hydrolysis process that normally converts it to glucose and to attack by bacteria. The hydrous cellulose pulp now has an unlimited shelf life at normal temperatures and conditions.

This bacteria resistant hydrous cellulose pulp, which possesses an unlimited shelf life, may be produced either by recycling waste waxed paper, new waxed paper or by processing virgin vegetable constituents in the presence of wax during the emulsification phase of the defibering process.

As a result of this invention, non-paper industry uses have been developed for hydrous cellulose pulp possessing fibers coated with a thin micro-molecular layer of wax.

The fiber material derived from this process can be combined with products such as toothpaste, shampoo, soap, detergent and lotions or creams in liquid form such that the fiber particles become suspended evenly throughout the detergent and soap and greatly enhance the cleaning property of the detergent and soap.

The fiber material derived from this process can be used to produce a moisture retaining plant mulch that can be color coordinated with the plant.

The fiber material derived from this process can be used to simulate snow, from which realistic snow balls can be made in temperatures above freezing or the simulated snow can be used for skiing in warm climates.

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The micro-molecular film on the cellulose fiber filaments prevents decomposition and result in an unlimited shelf life. Furthermore, the filaments will not contaminate other materials to which they are added.

In accordance with this invention, an example of the starting waxed paper that can be used is the type used in bakeries and deli-contestants to wrap food products. Waxed paper of this type is coated with a food grade paraffin wax, designated as a dry wax. Waste waxed paper can be used in the preferred embodiment and is obtained directly from the paper producing facilities. For example, trimmings from a trimming machine or wax paper that did not meet required test standards may be used. Such waxed paper is free of printing and thus is clean. The waxed paper is added to a pulper. A pulper is basically a vat for receiving a material that can be agitated by mechanical means and includes means to control the temperature. The process of pulping is essentially one of separating cells from intercellular material. It should be understood that any equipment such as a conventional high speed pulper may be used. The temperature of the wax-containing fiber slurry is raised to a temperature above the melting point of the wax and beating is continued until the wax and fiber are released into the aqueous solution. The resulting water-fiber slurry can then be subjected to a washing process to remove any impurities. Newly manufactured waxed paper does not need this washing process.

The process of the present invention encompasses the use of 100% waxed paper stock having a wax content of up to 30% by weight. However, non-waxed waste paper, in modest proportions can be used without affecting the outcome. Non waxed fiber products can be used as a starting product and a paraffin wax in the correct ratio to fiber added. The use of waxed paper as a starting point has the advantage that it contains the proper ratio of fiber to wax and it is available at economical rates.

The water soluble non-ionic emulsifier that is added to the slurry being from the group consisting of: polyethylene glycol ethers of hydrophobic alcohols; alkylphenoxy polyethoxyethanols; fatty acid amides and mixtures thereof. The water soluble non-ionic emulsifier must also meet specific emulsion stability standards and depending upon the ultimate product it may be necessary that it comply with Food and Drug Administration requirements. The preferred water soluble non-ionic emulsifiers include: ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1; ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive; ethoxylated alkyl phenols in which the alkyl substituent is linear; and the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

After the hydrous cellulose pulp has been coated with the micro-molecular film, it can be mixed with products such as toothpaste, shampoo, soap, detergent and lotions or creams. Thus wax from the original waste waxed paper is present on the fiber filaments that are dispersed in the final product. However, such wax is present on the fibers filaments, in a finely divided form. The finely divided, dispersed wax on the fiber filaments

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in the detergent and soap product does not interfere with dispersion of the fiber in the liquid detergent and soap. In products such as liquid soap and shampoo the fiber filaments become suspended in the liquid and do not settle out. In products that are more viscous, such as a mouthwash or a glass cleaner the product must be treated, for example by running the product through a colloid mill, to maintain its suspension. When the fiber containing detergent and soap solution is used as a shampoo, for example, the dispersed fibers function as scouring elements that rub and massage the hair filaments to thereby greatly enhancing the cleaning properties of the detergent and soap solution.

In addition to shampoo this invention has been used as an additive to toothpaste, mouthwash, shaving cream, car washing detergents, dish-washing compounds, laundry detergents, industrial hand detergent and soap and liquid facial soap or lotion. In all of these applications the wax coated fiber filaments function to improve the effectiveness of the basic cleaning agent.

The resulting hydrous cellulose pulp material is stable and will not melt at temperatures above 32° Fahrenheit, is white in color and has a moisture content of about 90%. Simulated snow balls can be made from the hydrous cellulose pulp material by grasping a hand full of the material, packing it between the palms of the hands such that a small portion of the water is squeezed out and the resulting product can be thrown in the same fashion as a snow ball. There are children in parts of this country and in the world who have never had the opportunity to make and throw snow balls. A product for making simulated snow balls will be a fascinating toy for children in such regions. Also simulated snow can be used for skiing during the summer or for use at ski resorts when the temperature is above freezing.

A characteristics of the resulting hydrous cellulose pulp is its ability of retaining a high percent of water. A plant mulch has been developed from the resulting hydrous cellulose pulp that not only prevents the growth of weeds but is very beneficial in providing moisture for the plant over extended periods. Moisture from the mulch is absorbed by the soil at a rate that the moisture can be utilized by the plant. Since the hydrous cellulose pulp does not decompose, when the hydrous cellulose pulp becomes water depleted, it can be resaturated and the process repeated. Another advantage of this product is that the resulting hydrous cellulose pulp readily accepts dyes and thus can be dyed any of a desired colors. The resulting hydrous cellulose pulp has been dyed, Kelly green, and found to be a very attractive mulch for house plants. The Kelly green color being considered more attractive than the usual black or brown color of the soil. It is also contemplated that mulch could be produced in colors that are coordinated with the color of the flowers.

The following are examples of products which have been improved through the use of this invention. Shampoo-made in accordance with this invention has resulted in an improved product that leaves the hair feeling softer and having a brighter appearance. In addition the amount of hair that is collected in a hair brush that is used after grooming with the improved product is reduced. Furthermore shampoo made in accordance with this invention functions to detangle the hair and has been found to be particular beneficial when used on difficult to manage hair such as hair that is course and kinky.

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Shampoo that especially formulated for animals such as dogs and horses that has been improved in accordance with this invention has been found to be greatly improved especially because of its improved ability to detangle and soften the appearance of the hair.

Skin creams have been improved in accordance with this invention and conditions such as "ash" have been greatly improved or eliminated.

When toothpaste that has been improved in accordance with this invention is used, a marked reduction in bleeding of the gums and plaque build up has been noted.

When shaving creams are improved in accordance with this invention, closer shaves and a smoother shaven surface are obtained.

While the invention has heretofore been described in detail with particular reference to specific products, it is to be understood that variation, modifications and the use of equivalents can be effected without departing from the scope of this invention. It is, therefore, intended that such changes and modifications be covered by the following claims.

I claim:

1. A process for improving mouthwash, glass cleaner, toothpaste, shampoo, soap, detergent and lotions or creams comprising the steps of:

(a) producing a decomposition resistant hydrous cellulose pulp, the individual fibers of which are coated with a thin wax film;

(b) adding the decomposition resistant hydrous cellulose pulp to the product while the product is in liquid form;

(c) blending the resulting mixture such that the fibers of the decomposition resistance hydrous cellulose pulp are dispersed and suspended in the product where they function as scrubbing and massaging agents to enhance the cleaning and conditioning function of the product.

2. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

3. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

4. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsifier that meets the following emulsion stability standards is used:

ethoxylated alkyl phenols in which the alkyl substituent is linear.

5. The invention as set forth in claim 1 wherein in the process for producing the decomposition resistant hydrous cellulose pulp a water soluble non-ionic emulsi-

5,412,090

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fier that meets the following emulsion stability standards is used:

the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

6. Mouthwash, glass cleaner, toothpaste, shampoo, soap, detergent, lotions or creams having an additive dispersed and suspended therein, each fiber of the additive acting as a scrubbing and massaging agent as the product is applied to the surface to be cleaned or conditioned;

said additive being a decomposition resistant wax coated hydrous cellulose pulp.

7. The invention as set forth in claim 6 wherein said decomposition resistant hydrous cellulose pulp is made in a process in which a water soluble non-ionic emulsifier that meets the following emulsion stability standards was used:

ethoxylated aliphatic alcohols wherein the alcohol is a hydrophobic secondary alcohol having from 11 to 15 carbon atoms and wherein the average molar ratio of ethylene oxide to hydrophobic alcohol is in a range of 5:1 to 15:1.

8. The invention as set forth in claim 6 wherein said decomposition resistant hydrous cellulose pulp is made in a process in which a water soluble non-ionic emulsifier that meets the following emulsion stability standards was used:

ethoxylated alkyl phenols in which the ratio of moles of ethylene oxide per mole or ethylene oxide per mole of alkyl phenol is in the range of 7-8 inclusive.

9. The invention as set forth in claim 6 wherein said decomposition resistant hydrous cellulose pulp is made in a process in which a water soluble non-ionic emulsifier that meets the following emulsion stability standards was used:

ethoxylated alkyl phenols in which the alkyl substituent is linear.

10. The invention as set forth in claim 6 wherein said decomposition resistant hydrous cellulose pulp is made in a process in which a water soluble non-ionic emulsifier that meets the following emulsion stability standards was used:

the fatty acid amide diethanol amine condensates derived from a member selected from the group consisting of myristic acid, lauric acid, palmitic acid, stearic acid and mixtures thereof.

11. A method of producing simulated snow from hydrous cellulose pulp material that has high water retention, that is stable and has an unlimited shelf life comprising the steps of:

a) providing a hydrous cellulose pulp material comprising fibers and having a water content of about 90%; and

(b) covering each fiber of said hydrous pulp material with a thin wax coating to prevent decomposition thereof.

12. The method as set forth in claim 11 wherein said simulated snow has the additional property that a portion of its water content can be squeezed out to simulate snow being packed.

13. A method of producing a mulch for plants from hydrous cellulose pulp material that has high water retention, that is stable and has an unlimited shelf life comprising the steps of:

(a) providing a hydrous cellulose pulp material comprising fibers and having a water content of about 90%; and

(b) covering each fiber of said hydrous cellulose pulp material with a thin wax coating to prevent decomposition thereof.

14. The method as set forth in claim 13 wherein said mulch for plants is dyed to an earthen color or a color that coordinates with plants for which it is functioning as a mulch.

\* \* \* \* \*

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1/3,LS/1 (Item 1 from file: 654)

02409721

Utility

**HYDROUS CELLULOSE PULP FOR NON PAPER PRODUCTS**

[ Adjuvant for toothpaste, mouthwash, shampoo, mild abrasive, artificial snow, mulch for plants ]

**Patent Number:** 5,412,090

ISSUED: May 02, 1995 (19950502)

**Inventor:**

Bendiner Bernard 8815 W. Golf Rd., Suite 12D Niles IL (Illinois) US (United States of America)  
60714

[Assignee Code(s): 68000]

**Extra Information:**

Assignment transaction [Reassigned] recorded December 15, 1994 (19941215)

**POST-ISSUANCE ASSIGNMENTS**

ASSIGNEE(s): BENDINER, MATTHEW K. 1506 SWORDFISH WAY KITTY HAWK, NORTH CAROLINA 27947

Assignor(s): BENDINER, BERNARD -- signed: 03/31/1994

Recorded: December 15, 1994 (19941215)

Reel/Frame: 007205/0571

Brief: ASSIGNMENT OF ASSIGNOR'S INTEREST

Rep.: WILLIAN BRINKS HOFER GILSON & LIONE F. DAVID AUBUCHON P.O. BOX 10395 CHICAGO, ILLINOIS 60610

ASSIGNEE(s): NATURAL FIBERS, LTD. SUITE 12A 8809 WEST GOLF ROAD NILES, ILLINOIS 60714

Assignor(s): BENDINER, MATTHEW K. -- signed: 12/12/1994

Recorded: December 15, 1994 (19941215)

Reel/Frame: 007205/0574

Brief: ASSIGNMENT OF ASSIGNOR'S INTEREST

Rep.: WILLIAN BRINKS HOFER GILSON & LIONE F. DAVID AUBUCHON P.O. BOX 10395 CHICAGO, ILLINOIS 60610

ASSIGNEE(s): PRESERVATION PRODUCTS, INC. 326 WOODSEdge, SUITE B MICHIGAN CITY, INDIANA 46360

Assignor(s): NATURAL FIBERS, LTD. -- signed: 04/29/1999

Recorded: May 3, 1999 (19990503)

Reel/Frame: 009942/0357

Brief: ASSIGNMENT OF ASSIGNOR'S INTEREST

Rep.: BRINKS HOFER GILSON & LIONE F. DAVID AUBUCHON P.O. BOX 10395 CHICAGO, IL 60610

**Application Number:**

8-190,301

FILED: February 02, 1994 (19940202)

Full Text: 371 lines

US PAT.FULL. (Dialog® File 654): (c) format only 2001 The Dialog Corp. All rights reserved.

ASSIGNMENT

WHEREAS, NATURAL FIBERS, LTD., a corporation organized and existing under the laws of the State of Illinois, having a place of business at 8809 West Golf Road, Suite 12A, Niles, Illinois 60714, hereinafter called the "Assignor", is the Assignee of the invention described in the United States patent entitled HYDROUS CELLULOSE PULP FOR NON PAPER PRODUCTS, for a full description of which reference is here made to Letters Patent of the United States No. 5,412,090 issued on May 2, 1995;

WHEREAS, PRESERVATION PRODUCTS, LLC, a corporation organized and existing under the laws of the State of Indiana having a place of business at 326 Woodsedge, Suite B, Michigan City, Indiana 46360, hereinafter called the "Assignee", desires to acquire the entire right, title and interest in and to the invention and the Letters Patent identified above;

NOW, THEREFORE, in consideration of the sum of One Dollar (\$1.00), and other valuable and legally sufficient consideration, the receipt of which by the Assignor from the Assignee is hereby acknowledged, the Assignor has sold, assigned and transferred, and by these presents does sell, assign and transfer to the Assignee, the entire right, title and interest for the United States in and to the invention and the Letters Patent identified above, and all patents that may issue for said invention in the United States; together with the entire right, title and interest in and to said invention and all Letters Patents therefor in all countries foreign to the United States, including the full right to claim for any such application all benefits and priority rights under any applicable convention; together with the entire right, title and interest in and to all continuations, divisions, renewals and extensions of any of the Letters Patents defined above; to have and to hold for the sole and exclusive use and benefit of the Assignee, its successors and assigns, to the full end of the term or terms for all such patents.

The Assignor hereby covenants and agrees, for both the Assignor and the Assignor's legal representatives, that the Assignor will assist the Assignee in the making and prosecution of any other patent applications that the Assignee may elect to make covering the

EXHIBIT


H

Case: 1:01-cv-07403 Document #: 1 Filed: 09/25/01 Page 46 of 59 PageID #: 46  
invention identified above; in vesting in the Assignee like exclusive title in and to all such  
other patent applications and patents; and in the prosecution of any interference which may  
arise involving said invention, or any such patent application or patent; and that the Assignor  
will execute and deliver to the Assignee any and all additional papers which may be  
requested by the Assignee to carry out the terms of this Assignment.

The Commissioner of Patents and Trademarks is hereby authorized and requested to  
issue patents to the Assignee in accordance with the terms of this Assignment.

IN TESTIMONY WHEREOF, the Assignor has executed this agreement.

DATED: April 29, 1999  
\_\_\_\_\_  
\_\_\_\_\_

  
\_\_\_\_\_  
Bernard Bendiner  
\_\_\_\_\_  
Natural Fibers, Ltd.  
\_\_\_\_\_

STATE OF ILLINOIS )  
 ) ss.  
COUNTY OF COOK)

I, Arlette Porter-Sherley, a Notary Public in and for the County and State aforesaid, do hereby certify that Bernard Bendiner, of NATURAL FIBERS, LTD., known to me to be the same person whose name is subscribed to the foregoing instrument, did sign, seal and deliver the said instrument as his free and voluntary act for the uses and purposes therein set forth.

IN WITNESS WHEREOF, I have hereunto set my hand and Notarial Seal, this 29<sup>th</sup> day of April, 1999.



Arlette Porter Sherley  
Notary Public

My Commission Expires: June 25, 2001

Welcome to NCLII!

# Welcome to NCLII!

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- Facilities
- Products
- Services
- News & Updates
- Subsidiaries
- About our Founder



**Welcome to**  
**Nutraceutical Clinical Laboratories International, Inc.**

*Please take a minute to read our*

### Executive Summary

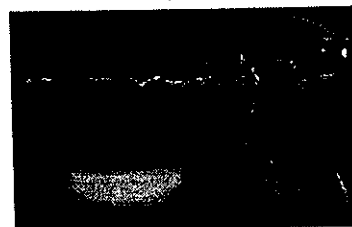
Nutraceutical Clinical Laboratories International, Inc. was founded in September 1997 to play a pivotal role in the legalization of nutritional, nutraceutical, and alternative health formulations.

This industry is growing faster than any other industry despite the lack of regulations. The reason for the rapid growth rate is that many of these supplements are safe and effective and do treat, mitigate, and even cure disease better than many of the so-called "ethical" drugs approved by the Food & Drug Administration (FDA) under the food, drug, and cosmetic regulations.

The FDA has determined that if the nutraceutical industry is to realize its full potential, then it must find a way to enable manufacturers to prove, through actual human testing, their stated beneficial claims. In the eyes of the federal regulators, there are many facets to be considered in determining the most expeditious and economical path to legitimacy. The problems have been enumerated by the news media; and, the networks have aired exposes on issues that range from contaminated dosages to variances in label claim content. These labels may even include false claims or claims that cannot be proven.

As a result of the recent developments in the nutraceutical products arena, an all natural food preservative would be required.

It will now become our major focus.



**EXHIBIT**  
**I**



# Liquisorb

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**LIQUISORB is an all natural Preservative for Food and Beverages (COMMERCIAL BRAND NAME).**

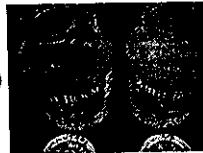
The product is designed to extend the shelf life of many processed foods and beverages from 3 to 5 times longer than the present chemical preservatives. Although it does replace chemical preservatives and provides extended life, the greatest advantage is to preserve freshness after opening.

***Foods that normally spoil within a few days after opening can now be preserved for weeks.***

- ***This is not an anti-microbial solution***
- ***It is not a sanitizer***
- ***It is not designed to replace pasteurization or sterilization***
- ***It does not replace good sanitation habits or procedures in the fill process***

**LIQUISORB is designed to replace chemical preservatives without increase in cost. It eliminates the need for adding flavors and fragrances to cover up the taste or odor created by chemical preservatives.**

**View for yourself the possibilities:**

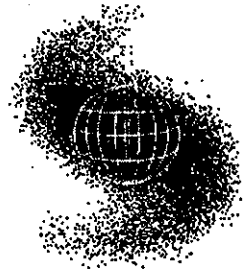


Applesauce that is exposed to harsh environmental conditions, can last up to 16 months unrefrigerated.



Fish filets, normally good for 3-5 days, when treated with LIQUISORB remained fresh and safe for 24 days—that's 5 times longer!

The development of this revolutionary new preservative product opens the door for hundreds of new foods and beverages, including new protein drinks and sport drinks. Savings on the preservative costs alone related to the Food and Beverage Industry could exceed hundreds of millions of dollars per year, as well as the savings gained from the prevention of spoilage. Also the increased revenues and profits associated with the sales of previously unavailable foods and drinks to millions of customers is another consideration.



We believe that our product will out perform all other preservatives on the market today. Our high-quality manufacturing processes, equipment, and testing laboratories provide you with an innovative, cost-effective technology that will result in immeasurable benefits.

Send mail to [webmaster@nutradata.com](mailto:webmaster@nutradata.com) with questions or comments concerning this web site.

Thank you for visiting our web site. Those who rely on this site for investor information are advised to view the updates prior to making investment decisions.



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Nutraceutical Clinical Laboratories International, Inc.  
Last modified: May 08, 2001

## Sampling Policy

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## SAMPLING AND TESTING POLICY

NCLII's products and services are unique and must be treated as such. Our preservative is very unique, and presently there are no standard testing procedures in the industry for evaluating the efficacy of the product.

NPTI has developed test procedures for specific products based on several key factors:

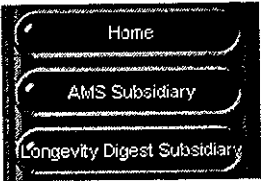
- Samples are released on a case by case basis for efficacy testing
- In each case, test protocols for those specific products will accompany the samples along with an agreement that all tests will be conducted in accordance with those protocols
- Our company will gladly review other protocols and will approve the same if the protocols are designed to test the products within the limits of our claims
- To protect unauthorized use of the test samples, we use the same "Chain of Custody" for samples required by the FDA for independent submissible test programs
- The necessary "Chain of Custody" forms will be supplied once the tests protocols have been agreed upon in writing.
- Each sample will have batch and quality control traceability

This policy is not new or unique and is used in virtually all submissible studies in the pharmaceutical market under the same regulations that NPTI currently uses.



# Subsidiaries

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In order to round out the marketing strategy for the companies products, several acquisitions appeared to be the fastest and mostly effective route for achieving our goals.

With that in mind the company began over two years ago to seek out and buy other entities that had the synergy needed to accomplish the procurement of the proper avenues for our marketing strategy.

We acquired 56.2% of American Medical Specialties Inc. AMS distributes Surgical blades and Burs for the surgical and dental markets and has over 50 independent reps calling on the majority of the Hospitals, surgical centers and dental labs around the US. NCLII has 5 patented products to be marketed to the same market the same customers as those of AMS. With the introduction of those products the company believes that it can double the sales and boost the profits of an already profitable subsidiary.

The company then purchased 100% of the stock in a magazine by the name of "Texas Fitness and Longevity Digest." This publication was purchased to reach the aging crowd with more than 30 products in the Nutraceutical and anti-aging category. The publication is expected to debut on the world wide web early in the Fall 2001 and move on to the national distribution through news stands by the first quarter of 2002.

The publication will focus on the aging process and provide useful suggestions for those concerned about the aging. Where at all possible NCLII provide products and services relating to the subject material.

Natural Preservation Technology Inc. is a wholly owned subsidiary of the parent company, NCLII and provides contract production for a variety of Nutraceutical products. NPTI also produces products distributed by NCLII and manufactures the liquid preservative for the food and beverage industry. The company's R&D Laboratories are located in the 37,000 sq ft NPTI facility.



NPTI Office

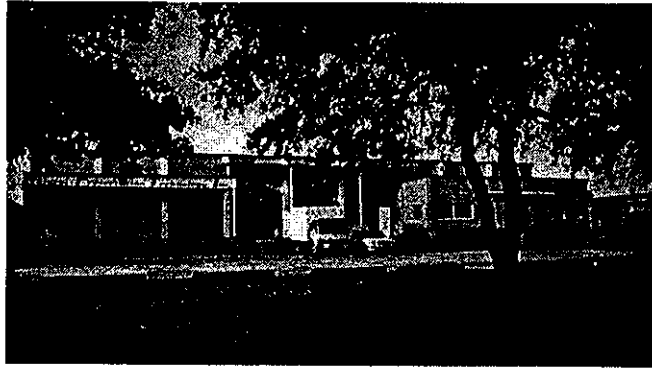
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NPTI News

The *Offices* of Natural Preservation Technology, Inc.



2440 30th Ave. N.  
St. Petersburg, FL 33713 USA  
727-526-9500  
FAX 727-823-1086

NPTI is a wholly owned subsidiary of NCLII and is housed in a 37,000 square foot facility just a few blocks away from the corporate headquarters. After extensive development, an innovative and all natural preservative, named NuPreserv, was created. However, it soon became evident that there was a much larger market for the preservative. Consequently, NPTI was acquired to manufacture NuPreserv for NCLII.

Equipment

Facilities

Nutritional Chemistry



Microbiology Laboratory

NUTRACEUTICAL CLINICAL LABORATORIES INTERNATIONAL, INC.

Summer 2001  
Volume 1, Issue 1



## THE NCCL SHAREHOLDER REVIEW

### Full Production Capability Reached At 30th Avenue Plant!

**Inside this issue:**

Management Expansion

Meet Betty

Start Production!

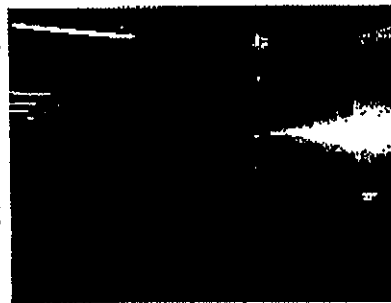
New Product Line

More Patents

New Director

Longevity Magazine

After many delays brought on by the re-zoning process and scale up problems, the first preservative line is complete and we now have full



30th Avenue Production Facility

production capabilities. Prior to starting the construction of the lines in future facilities, we have begun the process of automating and computerizing the line. Our plans include the expansion of the number of facilities in order to have

one within 400 miles of each major customer. The cost of each new facility has been estimated at one million

dollars and will have the capability of producing 24,000 gallons per day, per line. Four facilities are anticipated at this time. Each plant will include a quality control laboratory and production lines similar to the one in St. Petersburg.

**Special points of interest:**

- Dr. Richard Goldfarb Expected To Join The Board Of Directors
- NCLII Is Building Financial Stability
- NCLII Plans To File Two More Patents By Year End
- Longevity Magazine To Debut Online Before Nationwide Newsstand Roll-Out

### LIQUISORB Inventory Ready For Immediate Delivery

As of July 1, 2001, we have inventoried product for immediate delivery in 60 gallon drums for those users who do not need tank truck quantities. We can serve customers that are located within 400 miles of the St. Petersburg facility. Finally, the permitting process for our plant is near completion and we are in the process of completing the

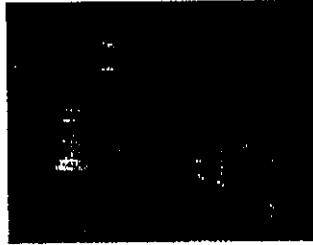
nutraceutical portion of the facility on 30<sup>th</sup> Avenue. Our capsule machines, tablet press, and blender are on site. Raw materials are on hand for the first capsule and tablet orders which we have at this time. We expect to slowly ramp up this division of our firm since our primary focus is the Preservative side of the business.

EXHIBIT

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## New Line Of Patented Infection Control Products To Be Introduced By American Medical

The prime reason for the acquisition of AMERICAN MEDICAL SPECIALTIES, INC., other than acquiring a quality company, was to obtain a marketing vehicle for six of the products patented by our founder, Paul Simmons, more than 10 years ago, along with some new ones developed in the past 24 months. We plan to begin marketing to hospitals, surgery centers, and dental offices the world's first and only "INSTRUMENT PRE-SOAK" that neither rusts nor damages sensitive dental and surgical instruments. Because of the absence of competition, we hope to have this



AMS Instrument Pre-Soak

product in every hospital, surgical center, and dental clinic in America. Other new products include a non-toxic surface disinfectant, a surgical scrub, a hand sanitizing gel, a first aid spray, and a patient pre-op scrub. AMS has been profitable for the past four years

and is growing at a rate of 20% per year. NCLII expects to double the sales of AMS during the next 18 months due to the addition of these products. The Company plans to register these products with the EPA in each state. They have already been approved by the U.S. EPA and FDA where required.

## NCLII Continues To Seek New Patents



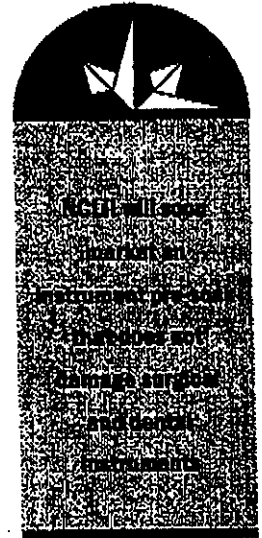
Patent Protection!

During the month of June, our Patent Attorney notified us that his preliminary search indicated that the two additional patents the Company sought are in his opinion, Patentable. NCLII immediately authorized him to

proceed with the necessary filings.

Keeping ahead of the technology is a key component to our future success.

It is likely that the company will file at least two more patents before the end of the year.



## Dr. Richard Goldfarb To Join The Board Of Directors Of NCLII

Subject to approval of the Board of Directors, Dr. Goldfarb will become a Director of NCLII. Dr. Goldfarb is a recognized pediatric surgeon from the Philadelphia area. He has been working with the company on a consulting basis, primarily in the clinical facility planning area. He has also been involved in the planning for the expansion of American Medical Specialties Inc.

We welcome Dr. Goldfarb to the board and look forward to his active participation.

## Production Facility Takes On A Natural Look

Keith Anderson, a member of the Board of Directors, and his crew decided to spruce up the plant with several innovative ideas following the natural theme. The photo displays one of three original 3-dimensional creations of Mr. Anderson and Donny Waters. Guests touring the facilities are enamored with the creativity and believe it lends to the all natural product theme of NCLII. Our thanks to both Keith and Donny for this unusual and beautiful creation.



Beautiful Interior Design

## Executives From Perrier & Toys"R"Us Join NCLL

After more than 20 years with Perrier Corporation, Frank de Vries, who retired at a very young age, has elected to join NCLL as the Vice President of Marketing and bring his marketing and contract negotiation skills to bear on this tremendous opportunity. With a worldwide need for the preservative product, NCLL is literally flooded with requests for more information from organizations around the globe. Until now, it has been impossible for us to follow through on all of these inquiries and to determine which we can serve and which are the most advantageous to the Company's overall plan for growth.



Frank de Vries & Keith Van Beek



to us a wealth of experience in managing companies this size as well as much larger ones. He is a tough negotiator and has many connections at the top of major prospective customer firms. If the relationship continues as it is today, we will present him to the Board of Directors for approval as CEO in August.



Jim Capwill

Also, James Capwill, a licensed CPA, has been working with us for the past several months as a consultant and has been placed in charge of our first certified financial audit. Assuming our mutual evaluation indicates a good fit, Jim will be presented the Board of Directors as a candidate for CFO in August. This is another step in NCLL's effort to build an elite management team in order to fully realize the potential of the opportunities before us.

Keith Van Beek is working out of our corporate headquarters in a consulting capacity, while we decide whether or not he and NCLL are a good match. Keith brings

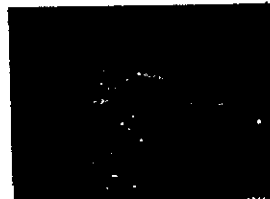
## Funding Situation Improving With Increased Production!

As most of you as shareholders know, financing has severely hampered our growth. Long delays brought on by the City of St. Petersburg in the zoning and permitting process postponed our plans for more than nine months. Investors want to see production and when that is

impeded, their interest wanes. Now that production is possible in both sides of our facility, interest has increased and the availability of funding has improved. We now have the necessary funds to complete the facility and to purchase the essential raw materials.

## Want To Place A Face With The Nice Lady At The Front Desk?

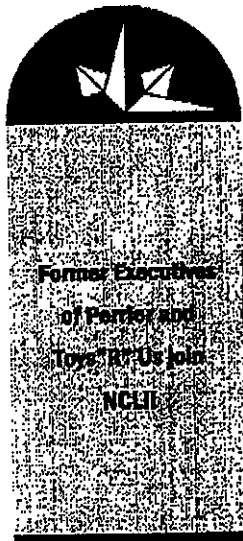
Betty Estes has been with the company for the past several months and is our link with the telephoning public. Many of you have gone out of your way to compliment Betty on the efficient way she handles calls.



Say Hello To Ms. Betty Estes!

As most of you who call in to the corporate offices, know, Betty is a busy person handling over 200

calls a day. We have received some complaints about the computer answering the telephone during the day. This only occurs when our switch board has reached its momentary capacity. When this happens you may leave a message or try your call again in five minutes. We work hard to keep this inconvenience to a minimum.





Nutraceutical Clinical  
Laboratories International,  
Inc.

NCLII Corporate Offices  
3542 Morris Street North  
St. Petersburg, FL 33713  
USA

Phone: 727-526-9500  
Fax: 727-527-4827  
Email: info@nutradata.com



**NCLII**

PRESERVING  
THE WORLD.

We're on the Web!  
[www.nutradata.com](http://www.nutradata.com)

Nutraceutical Clinical Laboratories International, Inc. was founded in September 1997 to play a pivotal role in the legalization of nutritional, nutraceutical, and alternative health formulations. The NCLII Corporate Offices are located on 3542 Morris Street North, St. Petersburg, Florida. The NuPreserv™ manufacturing facility is located a few blocks away at 2440 30<sup>th</sup> Avenue North. American Medical Specialties, Inc. is located near the corporate offices.

Mr. Paul L. Simmons is the Chairman of the Board and CEO. The Company currently trades on the Pink Sheets, LLC, "Pink Sheets", under the ticker symbol "NCCL".

For additional information, please contact our Vice President of Shareholder Relations, Ms. Pat Koepke.

*Our SHAREHOLDERS Are Our VIP's, THANK YOU!*

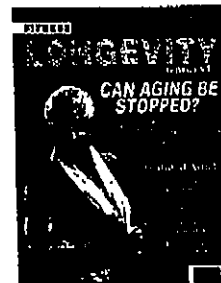
### New R&D (Bakery Products) Laboratory To Be Installed in 30th Ave. Manufacturing Facility

The Bakery Industry is one of the markets which stands to gain the most from the use of the LIQUISORB preservative. Laboratory tests indicate that bread can be preserved for weeks, or even months, beyond its present shelf life. Currently, there is a need to extend the shelf life for an extra 7 days. An additional 7 days could easily cut the costs of the products by making delivery to the stores once a week rather than twice a week, saving 50% in the distribution cost alone. Within the next 30 days, we expect to complete the laboratory, to hire some specialists from the baking industry, and conduct extensive studies using "state of the art" equipment and the latest technology. Any one of the major players in the baking industry could easily use one line of production capacity, giving them a major competitive advantage over the other baking firms.

### Longevity Magazine To Be Published Online Before National Newsstand Rollout

After many setbacks and false starts, *INTERNATIONAL FITNESS & LONGEVITY DIGEST* plans to go *live* on the Internet as a forerunner to national newsstand distribution. We expect to begin with a 60-page publication focusing on longevity and the anti-aging theme in the near future. When we go "live", visitors may view the site by simply logging on to

"www.longevitymagazine.com"  
We believe that our approach to the aging process is unique and very comprehensive. We expect to offer information concerning items that have been determined to contribute to the aging process and offer, where possible, alternative information and products for those who are serious about curbing the aging process as much as present technology will permit. We hope that you are excited as we



Bob Delmontague

Civil Cover Sheet

*Cal*

UNITED STATES DISTRICT COURT  
NORTHERN DISTRICT OF ILLINOIS

Civil Cover Sheet

**01C 7403**

JUDGE ANDERSEN

This automated JS-44 conforms generally to the manual JS-44 approved by the Judicial Conference of the United States in September 1974. The data is required for the use of the Clerk of Court for the purpose of initiating the civil docket sheet. The information contained herein neither replaces nor supplements the filing and service of pleadings or other papers as required by law. This form is authorized for use only in the Northern District of Illinois.

MAGISTRATE JUDGE DENLOW

Plaintiff(s): PRESERVATION PRODUCTS, LLC

County of Residence: Cook County, Illinois

Plaintiff's attorneys:

Robert M. Winter  
Robbins, Salomon & Patt,  
Ltd.  
25 E. Washington, Ste.  
1000, Chicago, IL, 60602  
312-782-9000

Defendant(s): NUTRACEUTICAL CLINICAL  
LABORATORIES INTERNATIONAL, INC.  
NATURAL PRESERVATION TECHNOLOGY  
INC., PAUL SIMMONS

Larry L. Saret  
Michael, Best & Friedrich,  
LLC  
401 North Michigan Avenue,  
Suite 1900, Chicago, IL,  
60611  
312-661-2100

FILED-EDA  
SEP 25 PM 1:15  
DISTRICT COURT

DOCKETED  
SEP 26 2001

II. Basis of Jurisdiction: 3. Federal Question (U.S. not a party)

III. Citizenship of Principle

Parties (Diversity Cases Only)

Plaintiff:- N/A

Defendant:- N/A

IV. Origin : 1. Original Proceeding

V. Nature of Suit: 830 Patent

VI. Cause of Action: 35 USC §§1-376; 15 USC 1051 et seq. Defendants are infringing on four U.S. patents and one trademark and committing deceptive trade practices.

VII. Requested in Complaint

Class Action: No

Dollar Demand: Preliminary and permanent injunction

Jury Demand: No

VIII. This case IS NOT a refilina of a previously dismissed case.

Signature: \_\_\_\_\_

*Robert M. Winter*

Date: September 25, 2001

1-2

**UNITED STATES DISTRICT COURT  
NORTHERN DISTRICT OF ILLINOIS**

Eastern Division

In the Matter of

PRESERVATION PRODUCTS, LLC  
vs.  
NUTRACEUTICAL CLINICAL LABORATORIES  
INTERNATIONAL, INC., NATURAL PRESERVATION  
TECHNOLOGY, INC. and PAUL SIMMONS;

**010 7403**  
**JUDGE ANDERSEN**

Case Number:  
**MAGISTRATE JUDGE DENLOW**

APPEARANCES ARE HEREBY FILED BY THE UNDERSIGNED AS ATTORNEY(S) FOR:

**PRESERVATION PRODUCTS, LLC**

<b>(A)</b>		<b>(B)</b>	
SIGNATURE <i>Robert M. Winter</i>		SIGNATURE <i>Larry L. Saret</i>	
NAME Robert M. Winter		NAME Larry L. Saret	
FIRM Robbins, Salomon & Patt, Ltd.		FIRM Michael, Best & Friedrich, Ltd.	
STREET ADDRESS 25 East Wasington Street, Suite 1000		STREET ADDRESS 401 North Michigan Avenue, Suite 1900	
CITY/STATE/ZIP Chicago, IL, 60602		CITY/STATE/ZIP Chicago, IL, 60611	
TELEPHONE NUMBER (312) 782-9000		TELEPHONE NUMBER (312) 661-2100	
IDENTIFICATION NUMBER (SEE ITEM 4 ON REVERSE) 03122228		IDENTIFICATION NUMBER (SEE ITEM 4 ON REVERSE) 02459337	
MEMBER OF TRIAL BAR? YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>		MEMBER OF TRIAL BAR? YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>	
TRIAL ATTORNEY? YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>		TRIAL ATTORNEY? YES <input checked="" type="checkbox"/> NO <input type="checkbox"/>	
		DESIGNATED AS LOCAL COUNSEL? YES <input type="checkbox"/> NO <input type="checkbox"/>	
<b>(C)</b>		<b>(D)</b>	
SIGNATURE		SIGNATURE	
NAME		NAME	
FIRM		FIRM	
STREET ADDRESS		STREET ADDRESS	
CITY/STATE/ZIP		CITY/STATE/ZIP	
TELEPHONE NUMBER		TELEPHONE NUMBER	
IDENTIFICATION NUMBER (SEE ITEM 4 ON REVERSE)		IDENTIFICATION NUMBER (SEE ITEM 4 ON REVERSE)	
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DESIGNATED AS LOCAL COUNSEL? YES <input type="checkbox"/> NO <input type="checkbox"/>		DESIGNATED AS LOCAL COUNSEL? YES <input type="checkbox"/> NO <input type="checkbox"/>	

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SEP 26 2001

09 SEP 25 PM 1:00  
U.S. DISTRICT COURT