

VANTAGE SPECIALTY CHEMICAL
v.
CARGILL, INC.

IPR2023-00589

Oral Hearing June 11, 2024
Patent Owner's Presentation

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE

Overview

- The '245 Patent claims a method for ester-ester exchange of jojoba wax esters using lipases.
- The prior art, alone or in combination, fails to suggest using lipases to catalyze ester-ester exchanges in jojoba wax esters.
- Moreover, the prior art, alone or in combination, fails to suggest that using lipases to catalyze ester-ester exchanges in jojoba wax esters would result in improved properties such as increased oxidative stability index (“OSI”) and improved physical properties.

The Invention of the '245 Patent

Overview of the '245 Patent

United States Patent

(12) Patent No. Addy et al.

(10) Patent No. Addy et al.

(45) Date of Publication

(54) PROCESSES AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)

(72) Inventor: Jeff Addy, Chandler, AZ (US); Clay Addy, Chandler, AZ (US); James H. Bland, Chandler, AZ (US)

(73) Assignee: International Flora Technologies, Ltd., Chandler, AZ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 1138 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: 14/578,075

(22) Filed: Dec. 19, 2014

(65) Prior Publication Data

US 2016/0077349 A1 Jun. 23, 2016

(51) Int. Cl. C12P 7/64 (2006.01)

C12P 7/64 (2006.01)

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(52) U.S. Cl. C12P 7/64 (2006.01)

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(53) CPC C12P 7/64 (2006.01)

C12P 7/64 (2006.01)

(54) Field of Classification Search CPC C12P 7/64 (2006.01)

C12P 7/64 (2006.01)

See application file for complete search history.

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C12P 7/64 (2006.01)

C12P 7/64 (2006.01)

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Primary Examiner—Lillian W. Thompson

Assistant Examiner—Lillian W. Thompson

(74) Attorney, Agent, or Firm—Fish & Richardson P.C.

(57) ABSTRACT

Processes for transesterifying wax esters, including wax esters, may include providing a feedstock including wax esters, contacting the feedstock with a lipase, and catalytically transesterifying the feedstock with the lipase to form a transesterified product. An oxidative stability index (OSI) of the transesterified product may be greater than an OSI of the feedstock.

5 Claims, 4 Drawing Sheets

Specification includes a Sequence Listing.

Vantage Exhibit 1001

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SUMMARY

Implementations of processes for transesterifying wax esters may include: providing a feedstock including wax esters, contacting the feedstock with a lipase, and catalytically transesterifying the wax esters in the feedstock with the lipase to form a transesterified product. An oxidative stability index (OSI) of the transesterified product may be greater than an OSI of the feedstock.

Overview of the '245 Patent

The '245 Patent discloses unexpected results when enzyme catalyst (lipase) is used instead of a chemical catalyst



(12) United States Patent
Addy et al.

(10) Patent No.: US 11,245,250
(5) Date of Patent: 11/25/2015

(54) PROCESS AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES
(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)
(72) Inventor: Jeff Addy, Chandler, AZ (US); Clay Addy, Chandler, AZ (US); James H. Blevins, Chandler, AZ (US)
(73) Assignee: International Flora Technologies, Ltd., Chandler, AZ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 1138 days.
This patent is subject to a terminal disclaimer.

(21) Appl. No.: 14/578,075
(22) Filed: Dec. 19, 2014
(65) Prior Publication Data
US 2016/077349 A1 Jun. 23, 2016

(51) Int. Cl.
C12P 7/64 (2006.01)
C12C 8/00 (2006.01)
C12C 1/00 (2006.01)
(52) U.S. Cl.
C12P 7/64 (2013.01); C12C 1/00 (2013.01)
(58) Field of Classification Search
CPC C12P 7/6458; C12C 3/005
See application file for complete search history.

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5,249,504 B2 1/2013 Ponce et al. 6/09 n/a
6,762,148 B1 6/2014 Liu 5/06 n/a
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2014/0126589 A1 5/2014 Aune C12A 21/18

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2003-53726 A 11/2003
2007-524191 A 10/2007
Primary Examiner—Louise W. Humphrey
(74) Attorney, Agent, or Firm—10b, & 10c
(57) ABSTRACT
Processes for transacetylating wax esters may include providing a feedback including the feedback with a lipase, and transacetylating the wax esters with the lipase to form a transacetylated product. An index (OSI) of the transacetylated product may be used to determine the feedback.

5 Claims, 4 Drawing Sheets
Specification includes a Sequence Listing

TABLE 3

Components	Feedstock Composition	Chemical Catalyst Change	% Enzyme Catalyst	% Change
Tocopherols	0.04%	0.00%	0.04%	0.00%
Fatty Acid Methyl Esters	0.00%	3.10%	0.00%	0.00%
Free Fatty Alcohols	0.00%	1.80%	0.00%	0.00%
OSI (hours)	26.5	21.5	38.6	45.66%
Wax Ester Content	98.00%	93.10%	98.00%	0.00%
Wax Ester Distribution				

Overview of the '245 Patent

The '245 Patent discloses unexpected results when enzyme catalyst (lipase) is used instead of a chemical catalyst



US011248245B2

(12) **United States Patent**
Addy et al.

(54) PROCESSES AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)

(72) Inventors: **Jeff Addy, Chandler, AZ (US); Clay Daly, Chandler, AZ (US); James H. Brown, Chandler, AZ (US)**

Brown, Chandler, AZ (US)
(73) Assignee: **International Flora Technologies,**

(*) Notice: Subject to any disclaimer, the term of

This patent is subject to a terminal extension of 5 years under U.S.C. 154(b) by 1138 days.

(21) Appl. No.: 14/578,075

(22) Filed:	Dec. 19, 2014	Deluxe Publications Data
(/65)		

(65) **Prior Publication Data**
US 2016/0177349 A1 Jun. 23, 2016

(51)	Int. Cl.	
	<i>C12P 7/64</i>	(2006.01)
	<i>C11C 3/00</i>	(2006.01)
	<i>C11C 3/00</i>	(2006.01)

(52) *ClIC 3/10* (2006.01)
U.S. Cl.
CPC *C12P 7/6436* (2013.01); *ClIC 3/00*

(58) Field of Classification Search
CPC C12P 7/6436; C11C 3/00
(2013.01); C11C 3/10 (2013.01)

USPC 438
See application file for complete search history.

(29)

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Year	Age	Population	Prevalence
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2000	A	Hepburn	6,123,979
2013	B2	Pearce et al.	8,340,594

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2014/01/20 589 Al • 5 2014 Austic C12M 2
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JP	Year of birth	Year of death
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material. The results also indicate that the chemically catalyzed reaction destroys the original amount of tocopherols present in the feedstock, which reduces the OSI of the transesterified product. Surprisingly, however, the OSI of the enzymatically catalyzed transesterified product is greater than that of the feedstock. This result indicates that the enzymatic catalysis has an unexpected positive effect on OSI that is not explainable simply by the enzymatic catalyst preserving the natural tocopherols originally present in the feedstock during the reaction. The OSI was measured using

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Overview of the '245 Patent

The '245 Patent discloses unexpected results when enzyme catalyst (lipase) is used instead of a chemical catalyst



TABLE 5

Transesterified Product Type in Cream	Viscosity	Appearance
Chemically Catalyzed	120,900 cP	White, semi-solid phase
Enzymatically Catalyzed	78,585 cP	White, liquid phase, glossy

Table 5 indicates that the use of enzymatically catalyzed transesterified jojoba oil created a cream formulation that had a viscosity drop of nearly 35% from the viscosity of the chemically catalyzed cream. This data indicates that the enzymatically catalyzed transesterified jojoba oil has the functional effect of lowering the resulting product viscosity compared to chemically catalyzed product. The difference in

Overview of the '245 Patent

Interestingly, both 1, 3 lipases and lipases that operate using insertion of a triglyceride work equally well when used in jojoba ester transesterification reactions. This result is unexpected, as these lipases sterically interact quite differently with the molecules they are involved in catalyzing. Without being bound by any theory, it is possible that because a jojoba ester is primarily a long carbon chain without much molecular bending due to the ester group, both 1, 3 lipases and lipases that operate using insertion are able to interact sterically with the jojoba esters in ways that produce substantially the same catalytic effect. In other words, the long chain jojoba ester can fit into the opening in an insertion lipase and also slide into the active areas of a 1, 3 lipase. As this processing characteristic for lipase catalytic processing of jojoba wax esters is not a predictable result, this illustrates a unique aspect of processing jojoba wax esters using lipases.

United States Patent Addy et al.

(54) PROCESSES AND SYSTEMS FOR
CATALYTIC MANUFACTURE OF WAX
ESTER DERIVATIVES

(71) Applicant: International Flora Technologies,
Ltd., Chandler, AZ (US)

(72) Inventor: Jeff Addy, Chandler, AZ (US); Clay
Addy, Chandler, AZ (US); James H.
Barnes, Chandler, AZ (US)

(73) Assignee: International Flora Technologies,
Ltd., Chandler, AZ (US)

(*) Notice: Subject to any disclaimer, this term of this
patent is extended or adjusted under 35
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This patent is subject to a terminal dis-
claimer.

(21) Appl. No.: 14/578,075

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US 2016/077349 A1 Jun. 23, 2016

(51) Int. Cl.
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C12P 7/64 (2013.01); C12C 6/00 (2013.01)

(53) CPC
C12P 7/64 (2013.01); C12C 6/00 (2013.01)

(54) Field of Classification Search
C12P 7/64 (2013.01); C12C 6/00 (2013.01)

See application file for complete search history.

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C12C 21/18 4/2014

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Overview of the '245 Patent

The '245 Patent discloses lipases result in higher production for jojoba wax esters than triglycerides



US 11,248,245 B2

(12) United States Patent
Addy et al.

(10) Patent No.: US 11,248,245 B2
(45) Date of Patent: Feb. 15, 2022

WO 2019/080232 A1 12/19/19
WO 1999/01512 A1 12/19/99

(50) PROCESSES AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)

(72) Inventor: Jeff Addy, Chandler, AZ (US); Clay Addy, Chandler, AZ (US); James H. Brown, Chandler, AZ (US)

(73) Assignee: International Flora Technologies, Ltd., Chandler, AZ (US)

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C12C 1/10 (2006.01)

(52) U.S. Cl.
C12P 7/64 (2006.01)
C12C 1/00 (2006.01)
C12C 1/10 (2006.01)

(58) Field of Classification Search
CPC C12P 7/6458; C12C 3/005
CPC C12P 7/6458; C12C 3/005
See application file for complete search history.

(59) References Cited

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5,249,504 B2 1/2013 Petro et al.
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435,134

FOREIGN PATENT DOCUMENTS

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2007-52491 A 10/2007

claiming the process which is characterized by the use of a lipase to form a transesterified product. An oxidative stability index (OSI) of the transesterified product may be greater than an OSI of the feedstocks.

5 Claims, 4 Drawing Sheets

Specification includes a Sequence Listing.

Vantage Exhibit 1001

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product to kg of catalyst ratio of 5011. This ratio of kg of product to kg of immobilized catalyst is over 20% greater than the ratio when the same lipase is used for triglyceride production. This result when the lipase is used for catalytic transesterification of jojoba oil significantly exceeds the conventional expectations for the lipase, and is an unexpected result that is not predicted by the results of conventional enzymatic triglyceride processing. In various imple-

Prosecution History of the '245 Patent

Applicant distinguished invention from alcoholysis reactions

PROCESSES AND SYSTEMS FOR CATALYTIC DERIVATIVES

BACKGROUND

1. Technical Field
[0001] Aspects of this document relate generally to processes and systems for catalytic derivatives and processing wax esters and wax ester derivatives.
2. Background Art
[0002] A wax ester is formed from the chemical reaction of an alcohol and a carboxylic acid. The alcohol, which results in the formation of an ester group, is found in various animals and plants, including bees, fish, and insects. Wax esters are used in various applications, including as lubricants, coatings, and in the production of various products.

<i>Applicant-Initiated Interview Summary</i>	Application No. 14/578,075	Applicant(s) Addy et al.		Page 1 of 2
	Examiner SRIKANTH PATURY	Art Unit 1657	AIA (First Inventor to File) Status Yes	

Issues Discussed:

35 U.S.C. 103

Applicant representative, attorney Whelan summarized that the instant invention is directed to a method comprising providing a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters and contacting the feedstock with a lipase to produce a transesterified product. Attorney Whelan argued that this increase of the OSI is unexpected and not suggested by the prior art. Attorney Whelan argued that the combination of Arquette and Steinke do not teach all of the elements of the method of claim 24, particularly that the feedstock comprises jojoba wax esters and hydrogenated jojoba wax esters and the amount of the hydrogenated jojoba wax esters is 20% to 50% by weight of the feedstock. Attorney Whelan argued that for the examiner to rely on inherency theory to support the obviousness rejection, the claimed property must necessarily result from combining the two references without modification and argued the 103 rejection should be withdrawn (also see attached interview agenda).

The examiners responded that the arguments are persuasive because the declaration filed on 07/20/2021 shows that the enzymatic transesterification of jojoba oil feedstock with a lipase results in significantly higher OSI even without the addition of an alcohol. The examiners agree that the increase in OSI is unexpected and not obvious over the prior art because Arquette and Steinke are silent about OSI and teach transesterification with a lipase with the addition of an alcohol to the feedstock. Therefore the rejections of claims 24-25 and 27-29 under 35 USC 103 over Arquette in view of Steinke will be withdrawn. The claims are free of the prior art.

Applicant distinguished invention from alcoholysis reactions

However, the prior art is silent about the OSI of the transesterified product and teaches transesterification of the feedstock with a lipase with the addition of an alcohol to the feedstock. The instant method wherein a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters is enzymatically transesterified with a lipase results in an unexpected higher OSI of the transesterified product as compared to the OSI of the feedstock even without the addition of an alcohol. Therefore, the instant method as recited in claim 1 is non-obvious over the prior art.

Prior Art Does Not Disclose That
Transesterification Increases OSI

Claim 1 of the '245 Patent

1. A process for transesterifying wax esters, the process comprising:
 providing a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters, wherein the amount of hydrogenated jojoba wax esters is 20% to 50% by weight of the feedstock;
 contacting the feedstock with a lipase; and
 transesterifying the jojoba wax esters and the hydrogenated jojoba wax esters in the feedstock with the lipase to form a transesterified product;
 wherein an oxidative stability index (OSI) of the transesterified product is greater than an OSI of the feedstock.

United States Patent

(12) Pat. No. 8,123,456

(63) Int. No. 14/578,075

(54) PROCESS AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)

(72) Inventor: Jeff Adley, Chandler, AZ (US); Clay Blythe, Chandler, AZ (US); James H. Blythe, Chandler, AZ (US)

(73) Assignee: International Flora Technologies, Ltd., Chandler, AZ (US)

(*) Notice: Subject to any disclaimer, this term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 1138 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No. 14/578,075

(22) Filed: Dec. 19, 2014

(65) Prior Publication Data US 2016/077349 A1 Jun. 23, 2016

(51) Int. Cl. C12P 7/64 (2006.01)

C12C 6/00 (2006.01)

(52) U.S. Cl. C12P 7/64 (2006.01)

C12C 6/00 (2006.01)

(58) Field of Classification Search CPC C12P 7/6456; C12C 3/003; C12C 6/003

See application file for complete search history.

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2014/0126289 A1 5/2014 Austin et al.

Specification includes a Sequence Listing.
 Vantage Exhibit 1001
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Vantage's Prior Art Fails to Teach or Suggest the Claimed Invention

- Vantage's Prior Art Does Not Disclose OSI of the Claimed Feedstock
- Vantage's Prior Art Does Not Disclose the OSI of the Claimed Transesterified Product.
- Prior Art Shows Results of Transesterification Was Not Predictable
- Because Number of Double Bonds Does Not Change, a POSITA Would Not Have Expected Increase in OSI

Cummings Does Not Disclose OSI of the Claimed Feedstock



POR, 35; EX1004, 2; Sur-reply, 3-5;
EX2009, ¶¶ 129-132



Cummings Does Not Disclose OSI of the Claimed Feedstock

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020
Issue Date: February 15, 2018
Appl. Serial No.: 14/578,075
Filing Date: December 11, 2014
Title: PROCESS
MANU-
DERIVAT

DECLARATION

I currently hold the op-
analysis may continue, and I
supplemental insights that may

I hereby declare that all s-
that all statements made on info-
declare that these statements
statements and the like so mad-
under Section 1001 of the Title

Dated: 12-6-23

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On the current record, we tend to agree with Patent Owner on this point. Elsewhere, Petitioner argues that Cummings’s “refined jojoba oil” forms only “part of” the feedstock. Pet. 38. We take that to mean the *unsaturated* part of the feedstock. And, if that is so, Petitioner does not identify clearly where Cummings discloses an OSI value of the fully saturated jojoba oil that forms the *other* part of the feedstock, much less the OSI of a feedstock mixture containing both the saturated and unsaturated parts. In other words, the OSI value of “around 35 hours” that Petitioner relies upon does not appear to reflect the OSI value of the actual feedstock mixture as a whole. See Ex. 1004, 2.

DI, 24; POPR, 8-12; POR, 40-42; Sur-reply, 10;
EX2009, ¶ 140-141

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 16

Brown Does Not Disclose OSI Values

Petitioner's Expert, Dr. Rockstraw:

David Rockstraw, Ph.D., P.E. – November 17, 2023

1 UNITED STATES PATENT AND TRADEMARK OFFICE
2 BEFORE THE PATENT TRIAL AND APPEAL BOARD
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5 VANTAGE SPECIAL CHEMICALS, INC.,
6 PETITIONER,
7 vs
8 CARGILL, INC.,
9 PATENT OWNER.
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Case No IPR2023-03
US Patent No 11,24

VIDEOTAPED HYBRID DEPOSITION
of
DAVID ROCKSTRAW, Ph.D., P.E.
New York, New York
Friday, November 17, 2023

Reported by:
Mary Agnes Drury, RPR, NYAOR, GLR

Q. Does Brown disclose OSI values?

A. No. My recollection is that Brown does not quantify changes in oxidative stability.

Q. So it doesn't disclose an OSI for the final product?

A. No. It does not provide quantitative values for oxidative stability.

Q. And it doesn't provide an OSI for the feedstock?

A. Not quantitatively, no.

Vantage's Prior Art Does Not Disclose the OSI of the Claimed Transesterified Product.

Inventor showed transesterification with a lipase resulted in three-fold increase in OSI (not shown in Cummings):

PROCESSES AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

1. Technical Field
- [0001] Aspects of this document relate generally to systems and processes for manufacturing wax ester derivatives.
2. Background Art
- [0002] A wax ester is formed from the chemical reaction of an alcohol, which results in the formation of an ester group that is covalently bonded to the alcohol. Wax esters are found in various animals and plants, including the cuticular layer of insects (e.g., bees, butterflies, etc.). Wax esters are used in various applications, including as lubricants, coatings, and in the manufacture of products.

2. This declaration outlines the results of an actual comparative experiment conducted between a chemically catalyzed transesterification of a mixture of hydrogenated jojoba oil (HJW) and refined jojoba oil and an enzymatically catalyzed transesterification of the same mixture like the experimental results disclosed in para. [0064] – [0070] of the present specification using 20% HJW. This experiment was conducted to further demonstrate the unexpected results of the

Type	AV	PV	DP	Lovi	OSI	IV
Feedstock	0.5	0.4	N/A	1.0R, 3.4Y	33.8	66.3
Enzymatic	0.19	0.9	48	0.9R, 2.9Y	117	65.7
Chemical	0.04	0.2	47.3	0.3R, 1.3Y	38	66.3

Table 1 -- Physical Properties

POR, 35; Sur-reply, 7; EX1002, 1074, 1076;
EX2009, ¶ 109

FISH

Cummings Does Not Disclose the OSI of the Claimed Transesterified Product.

Petitioner Relies on OSI Comparison of Commercial Products Containing OSI-Increasing Tocopherols



A natural alternative

Jojoba esters are a new category of naturally derived, oil free emollients that offer good properties for a wide variety of cosmetic products. Melanie Cummings, Kelley Dwyer and Robert Kliman of Floratex explain

Jojoba (*Euphorbia alata*) was first used by the Aztecs at least 1000 years ago. It is a shrubby plant that grows in the arid regions of Mexico and the southwestern United States. The Aztecs used the seeds for medicinal purposes and the leaves for dye. The plant is now grown commercially for its seeds, which are used to produce jojoba oil. This oil is a liquid wax ester, which is unique in that it is the only natural oil that is liquid at room temperature. It is also very stable and does not oxidize or become rancid. This makes it an ideal ingredient for cosmetic products. Jojoba oil is also known for its moisturizing properties and its ability to soothe dry skin. It is a natural alternative to many synthetic oils used in cosmetics.

Jojoba esters are a new category of naturally derived, oil free emollients that offer good properties for a wide variety of cosmetic products. Melanie Cummings, Kelley Dwyer and Robert Kliman of Floratex explain

Unique composition

While jojoba oil has unique and beneficial cosmetic properties its utility has been broadened significantly by using it as the base material for the creation of a new family of jojoba esters. Floraesters (INCI name: jojoba esters) are unique compositions of a broad range of saturated, partially saturated and unsaturated wax esters. Jojoba esters are made from 100% jojoba and were designed to vary in consistency from liquid to solid at ambient temperature. In the *CTFA Ingredient Handbook*, jojoba esters fall under the definition of a true ester and are not categorised within glyceryl esters, fats or oil families.

POR, 35-36; EX1004, 2; Sur-reply, 3-4; EX2009, ¶¶ 129-132

FISH

Cummings Does Not Disclose Claimed OSI Comparison

Jojoba Esters are proper waxes, with no triglyceride components. Jojoba Esters are a complex mixture of long chain (C35 to C46) fatty acids and fatty alcohols joined by an ester bond and do not contain any trans-unsaturation (International Jojoba Export Council 2004).

Floratech (2005a,b) reported the ester chain length and saturation of 4 Jojoba Ester products as shown in Table 6. Each was reported as being 99.95% Jojoba Esters and 0.05% tocopherol.

Simmondsia Chinensis (Jojoba) Seed Oil is combined with Hydrogenated Jojoba Oil and sodium methylate (catalyst) to get mixed Jojoba Esters (Floratech 2005a). Refined Simmondsia Chinensis (Jojoba) Seed Oil is combined with sodium methoxide to get randomized Jojoba Esters. Tocopherol is then added to make the commercial Jojoba Ester 15 (Floratech 2005b).

Final Report of the
Cosmetic Ingredient Review
Expert Panel

Safety Assessment of Simmondsia
Oil, Simmondsia Chinensis (Jojoba)
Hydrogenated Jojoba Oil, Hydrogenated
Isomerized Jojoba Oil, Jojoba Ester
(Jojoba) Butter, Jojoba Alcohol, and

The 2008 Cosmetic Ingredient Review Expert Panel members are: C. Donald V. Helito, M.D., Curtis D. Klassen, Ph.D., James G. Mark, Shiga, Ph.D., and Paul W. Snyder, D.V.M., Ph.D. The CIR Director is prepared by Lillian Becker, CIR scientific analyst.

Cosmetic Ingredient Review
1101 F Street, NW, Suite 415, Washington, DC 20004-2911, pp. 202-203

POR, 33-35; Sur-reply, 4-7; EX1028, 7;
EX2009, ¶¶ 140-141

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 20

Cummings Does Not Disclose Claimed OSI Comparison

Patent Owner's Expert, Dr. Schultz:

140. In my experience, a POSITA would instead have understood that ester products generally undergo additional processing after transesterification. Ex. 2064, 5. Specifically, with a jojoba wax ester, a POSITA would have understood that additional antioxidants (e.g., tocopherols) were likely to be added to increase OSI and enhance stability of the commercial product even further. It is the addition of tocopherols that explains why the OSI of refined jojoba oil is reported as 35 hours but the OSI of Floratech's Jojoba Esters 15 commercial product, which has the same number of double bonds as refined jojoba oil, is nevertheless reported as 100 hours (Ex. 1004, 2) or 70 hours (Ex. 1006, 3), respectively. Indeed, I understand that

IN THE UNITED STATES P

In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020
Issue Date: February 15
Appl. Serial No.: 14/578,075
Filing Date: December 11
Title: PROCESS
MANUFE
DERIVAT

DECLARATION

I currently hold the opi
analysis may continue, and I
supplemental insights that may

I hereby declare that all s
that all statements made on info
declare that these statements
statements and the like so mad
under Section 1001 of the Title

Dated: 12-6-23

34

A POSITA Would Not Have Expected Transesterification to Increase OSI

Patent Owner's Expert, Dr. Schultz:

138. In addition to failing to provide evidence showing the required comparison between the product and the feedstock, in my opinion Vantage's Petition also fails because it does not explain why a POSITA would have expected OSI to **increase** in comparison to the feedstock after transesterification, as required by claim

1. See Ex. 1001, cl. 1. In fact, a POSITA would not have expected OSI to increase after transesterifying jojoba wax esters, because the number of double bonds—i.e., the source of oxidative *instability*—does not change in the claimed ester-ester exchange. None of Vantage's other references say otherwise. Thus, Vantage has not met its burden to show any comparison, let alone that the results of such a comparison reveal an increased OSI in the transesterified product.

IN THE UNITED STATES PATENT

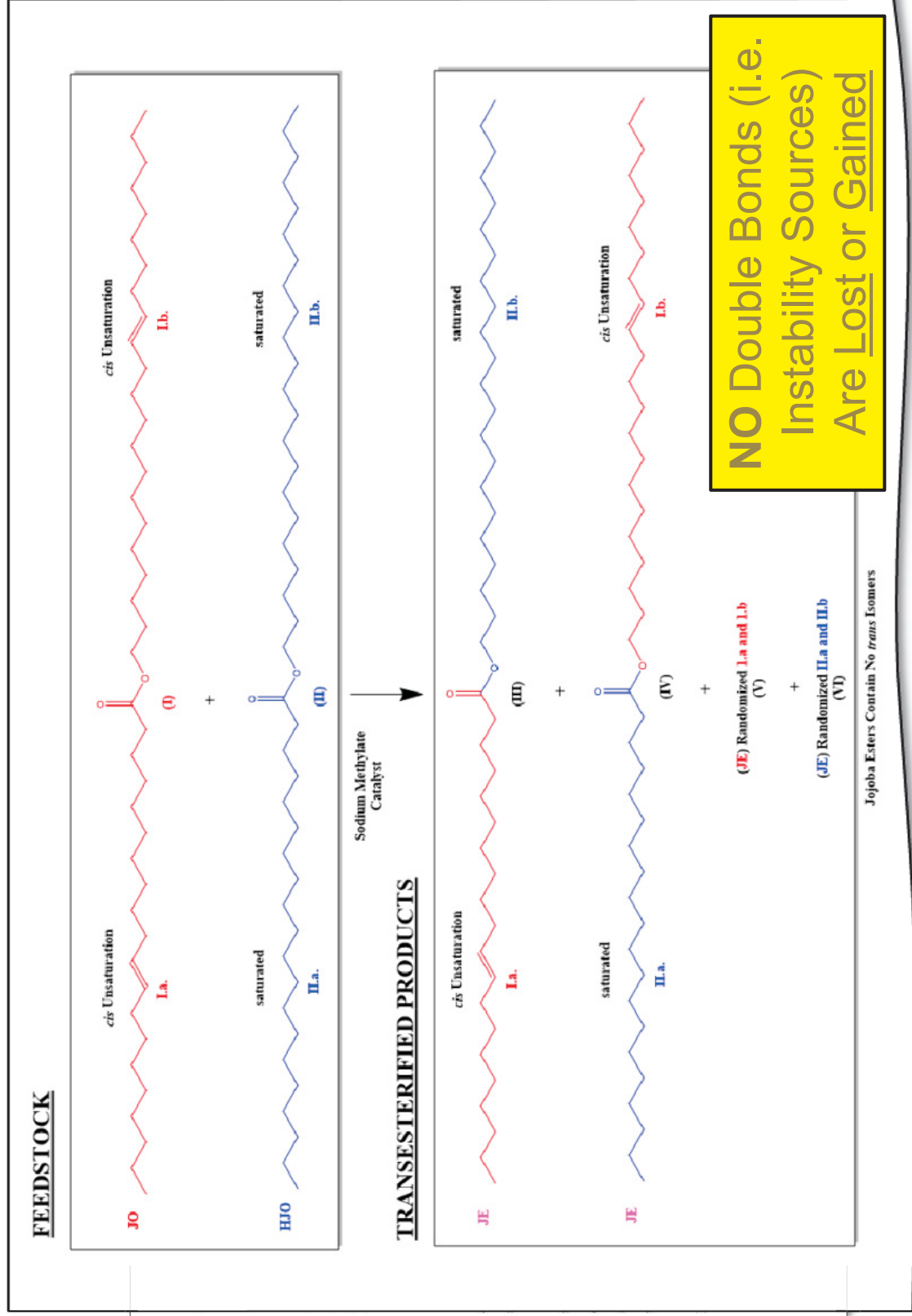
In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020IP1
Issue Date: February 15, 2022
Appl. Serial No.: 14/578,075
Filing Date: December 19, 2014
Title: PROCESS AND MANUFACTURE OF DERIVATIVES

DECLARATION

I currently hold the opinions and analysis may continue, and I may a supplemental insights that may result in I hereby declare that all statements that all statements made on information declare that these statements were in statements and the like so made are per under Section 1001 of the Title 18 of the

Dated: 12-6-23 JAA

A POSITA Would Not Have Expected Transesterification to Increase OSI



POR, 39-43; EX2009, ¶ 62 (adapted from EX1014, 6) DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 23

A POSITA Would Not Have Expected Transesterification to Increase OSI

Patent Owner's Expert, Dr. Schultz:

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020IP1

Issue Date: February 15, 2022
Appl. Serial No.: 14/578,075

Filing Date: December 19, 2014

Title: PROCESS AND SYSTEMS FOR CRYSTALLIZATION OF WAX DERIVATIVES

DECLARATION OF THOMAS SCHULTZ

I currently hold the opinions set expressed in this analysis may continue, and I may acquire additional information supplemental insights that may result in added observations.

I hereby declare that all statements made of my own knowledge that all statements made on information and belief are believed that these statements were made with the knowledge that statements and the like so made are punishable by fine or imprisonment under Section 1001 of the Title 18 of the United States Code.

Dated: 12-6-23 By: Thomas Schultz

In an ester-ester exchange between these two compounds, the fatty chains on either side of their ester bonds are swapped, randomizing the chains found in the mixture, such that there will be a reduction in the amount of fully saturated wax esters (zero double bonds) and unsaturated wax esters (two double bonds) coupled with an increase in the number of partially saturated wax esters (one double bond).

Critically, the total number of double bonds in the mixture *stays the same* before and after the ester-ester exchange. Ex. 2046, 80:19-22 (Dr. Rockstraw agreeing the

number of double bonds is the same in the feedstock and the product). As I explain

above, the number of double bonds in a compound is a key predictor of oxidative stability. Because there is no net gain or loss in double bonds in the claimed

transesterification process, in my opinion the POSITA would not have expected the oxidative stability to meaningfully change in the transesterified product.

A POSITA Would Not Have Expected Transesterification to Increase OSI

Chemical Transesterification Decreased OSI:

TABLE 3					
Components					
	Feedstock Composition	Chemical Catalyst	% Change	Enzyme Catalyst	% Change
Tocopherols	0.04%	0.00%	-0.04%	0.04%	0.00%
Fatty Acid	0.00%	3.10%	3.10%	0.00%	0.00%
Methyl Esters					
Free Fatty	0.00%	1.80%	1.80%	0.00%	0.00%
Alcohols					
OSI (hours)	26.5	21.5	-18.87%	38.6	45.66%
Wax Ester Content	98.00%	93.10%	-4.90%	98.00%	0.00%

The results also indicate that the chemically catalyzed reaction destroys the original amount of tocopherols present in the feedstock, which reduces the OSI of the transesterified product. Surprisingly, however, the OSI of the

A POSITA Would Not Have Expected Transesterification to Increase OSI

Commercial Products Added Tocopherols to Boost OSI:

Final Report of the
Cosmetic Ingredient Review
Expert Panel

Safety Assessment of Simmondsia Chinensis (Jojoba) Seed Oil, Simmondsia Chinensis (Jojoba) Seed Wax, Hydrogenated Jojoba Oil, Hydrolyzed Jojoba Esters, Isomerized Jojoba Oil, Jojoba Esters, Simmondsia Chinensis (Jojoba) Butter, Jojoba Alcohol, and Synthetic Jojoba Oil

September 23, 2008

The 2008 Cosmetic Ingredient Review Expert Panel members are: Chairman, Wilma F. Bergfeld, M.D., F.A.C.P.; Donald V. Beltrito, M.D.; Curtis D. Klaassen, Ph.D.; James G. Marks, Jr., M.D.; Ronald C. Shank, Ph.D.; Thomas J. Slaga, Ph.D.; and Paul W. Snyder, D.V.M., Ph.D. The CIR Director is F. Alan Andersen, Ph.D. This report was prepared by Lillian Decker, CIR scientific analyst.

Cosmetic Ingredient Review
1101 TP Street, NW, Suite 412 • Washington, DC 20005-4102 • Tel: 202-331-0667 • Fax: 202-331-0698 • ingredients@cir.org

Vantage Exhibit 1028
Page 1 of 37

Table 2. OSI of Jojoba Esters 15 and 60 when exposed to cosmetic actives (Brown et al. 1997)

Cosmetic active (%)	15 h	60 h
None	~35	~175
Tocopherols*	~80	~230
Iron oxides (10%) and tocopherols	~80	~225
Zinc oxides (10%) and tocopherols	~15	~90
Titanium dioxide (10%) and tocopherols	~135	~300
Malic acid (5%) and tocopherols	~110	~20
Salicylic acid (2%) and tocopherols	~25	~50
Salicylic acid (2%), titanium dioxide (10%), and tocopherols	~60	~180
Malic acid (5%), titanium dioxide (10%), and tocopherols	~120	~20
Arbutin (7%) and tocopherols	~50	~90
Kojic acid (1%) and tocopherols	~100	~250
Magnesium ascorbyl phosphate (3%) and tocopherols	~85	~170

* concentration of tocopherols not provided.

POR, 41; Sur-reply, 4-7; EX1028, 7;
EX2009, ¶¶ 140-141



A POSITA Would Not Have Expected Transesterification to Increase OSI

Commercial Products Added Tocopherols to Boost OSI:



TABLE 2. CHEMICAL AND PHYSICAL PROPERTIES OF JOJOBA ESTERS

Jojoba Ester	Melting Point °C	Iodine Value g I ₂ /100g	Oxidative Stability Value, Hours	Lubricity Degrees	Penetration, Mm	Monoene, Content, 0%
15	10-15	78-85	70	19.0	—	0
20	42-48	64-70	130	20.4	—	25-35
30	47-51	57-61	165	18.9	26.4	36-46
60	56-61	40-44	225	17.9	9.3	42-52
70	66-70	0-2	680	15.9	0.3	0



Vantage Exhibit Exhibit 1014
Page 1 of 8

POR, 41; Sur-reply, 4-7; EX1006, 3 (Tbl 2); EX1014, 7
(Tbl 2); EX2009, ¶¶ 140-141

FISH.

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 27

A POSITA Would Not Have Expected Transesterification to Increase OSI

Commercial Products Added Tocopherols to Boost OSI:

Certificates of Analysis

Jojoba Esters 15:

<u>Composition (INCI Names)</u>	<u>Wt. %</u>
Tocopherols	0.05
Jojoba Esters	99.95

Jojoba Esters 20:

Composition (INCI Names)		Wt. %
Tocopherols		0.05
Jojoba Esters		99.95

POR, 41; Sur-reply, 4-7; EX2012, 1; EX2013, 1; EX2009, ¶¶ 140-141

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 28



A POSITA Would Not Have Expected Transesterification to Increase OSI

11/10/20, 8:35 PM

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BUSINESS INSIGHTS: GLOBAL

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Trans Isomers in Cosmetics

Trans Isomers 1 cites to Rozenaal paper disclosing enzyme catalysts:

ALTERNATIVES TO TRANS ISOMERS IN COSMETIC PRODUCTS

The challenge of cosmetic ingredient suppliers is therefore to provide manufacturers with oxidatively stable and affordable lipid based emollients that exhibit a broad range of melting points yet which contain no trans fatty isomers. Oil phase emollients with these desirable characteristics can be obtained without the formation of trans isomers through a process known as "interesterification." Rozenaal [8] discussed use of this interesterification process to modify the melting characteristics of triglycerides using various catalysts including enzymes. Another

TRANS ISOMERS INHIBIT PROSTAGLANDIN SYNTHESIS

In vivo studies [1-2] using human skin cells have shown that some monoenes trans fatty acids inhibit both [delta]5 and [delta]6 desaturation of fatty acids. This synthesis pathway in humans to important prostaglandins require that the essential fatty acids, linoleic and linolenic acids, be first [delta]6 desaturated to [gamma]-linolenic and stearidonic acids respectively. These two acids are then elongated to their [C-sub.20] isomers. These isomers are

https://doi.org/10.1002/ajp.10005

1-5

Vantage Exhibit 1005

Page 1 of 5

A POSITA Would Not Have Expected Transesterification to Increase OSI

Petitioner's Expert, Dr. Rockstraw, Found But Did Not Cite Rozenaal:

David Rockstraw, Ph.D., P.E. – November 17, 2023

1 UNITED STATES PATENT AND TRADEMARK OFFICE
2 BEFORE THE PATENT TRIAL AND APPEAL BOARD
3
4
5 VANTAGE SPECIAL CHEMICALS, INC.,
6 PETITIONER,
7 vs
8 CARGILL, INC.,
9 PATENT OWNER.
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Case No IPR2023-03
US Patent No 11,24

VIDEOTAPED HYBRID DEPOSITION
of
DAVID ROCKSTRAW, Ph.D., P.E.
New York, New York
Friday, November 17, 2023

Reported by:
Mary Agnes Drury, RPR, NYAOR, GLR

Gregory Edwards, LLC | Worldwide Court Reporting
GregoryEdwards.com | 844-483-2643

Q. Did you look for that Rozenaal reference?

A. I did.

Q. Did you find it?

A. I did find it.

Q. Did you cite it on in your report?

A. I did not rely on it, so I did not cite it.

Q. Why didn't you rely on it?

A. Because the content that I needed was in Trans 1?

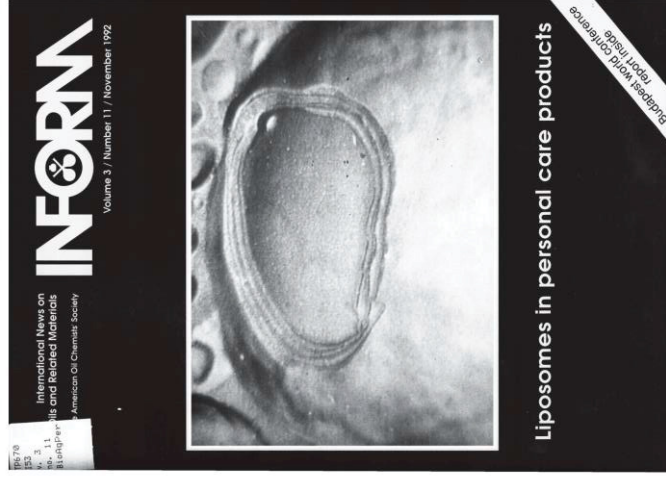
A POSITA Would Not Have Expected Transesterification to Increase OSI

Rozenaal is Independent, Contemporaneous Evidence That POSITA Expected OSI Not to Change During Interesterification (i.e. Transesterification):

Interesterification of oils and fats

Interesterification is one of three important fat modification processes.

Unlike hydrogenation, it leaves the fatty acid composition unchanged and only alters the distribution of the fatty acids over the triacylglycerols. The stability of the oil remains essentially unchanged, but important physico-chemical properties such as melting, crystallization and recrystallization behavior are modified.



CARGILL 2030
Vantage v. Cargill
IPR2023-00589

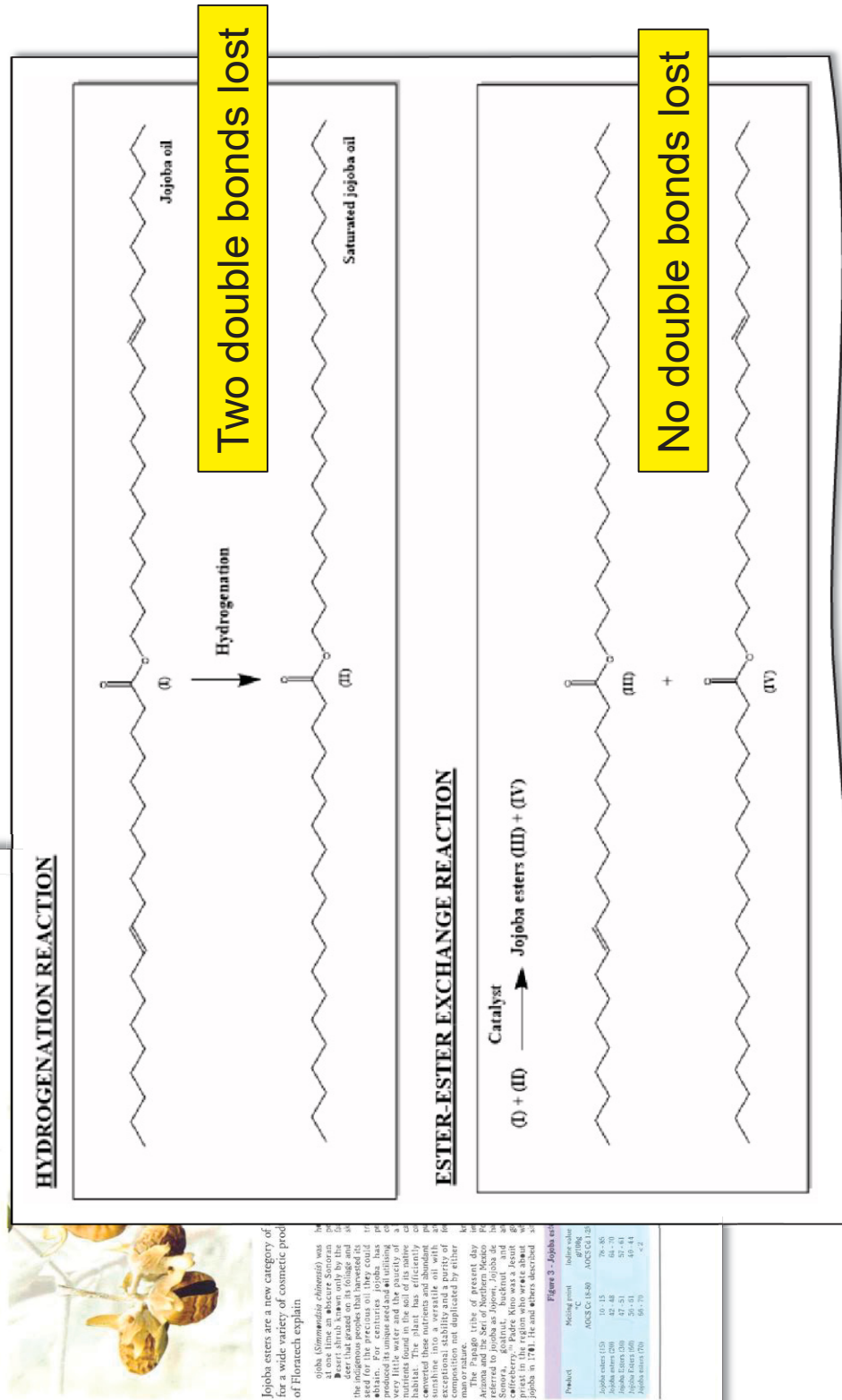
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FISH. POR, 43-44; EX2030, 3; EX2009, ¶ 144

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 31

A POSITA Would Not Have Expected Transesterification to Increase OSI

Cummings Discloses Both Hydrogenation (Increasing Saturation) and Transesterification (No Change in Saturation):



A POSITA Would Not Have Expected Transesterification to Increase OSI

Jojoba Esters 15 and 60 Both Contain Unsaturated Chains:

Final Report of the Cosmetic Ingredient Review Expert Panel

Safety Assessment of Simmondsia Chinensis (Jojoba) Seed Oil, Simmondsia Chinensis (Jojoba) Seed Wax, Hydrogenated Jojoba Oil, Hydrolyzed Jojoba Esters, Isomerized Jojoba Oil, Jojoba Esters, Simmondsia Chinensis (Jojoba) Butter, Jojoba Alcohol, and Synthetic Jojoba Oil

September 23, 2008

The 2008 Cosmetic Ingredient Review Expert Panel members are: Chairman, Wilma F. Bergfeld, M.D., F.A.C.P.; Donald V. Belsito, M.D.; Curtis D. Klaassen, Ph.D.; James G. Marks, Jr., M.D.; Ronald C. Shank, Ph.D.; Thomas J. Slaga, Ph.D.; and Paul W. Snyder, D.V.M., Ph.D. The CIR Director is F. Alan Andersen, Ph.D. This report was prepared by Lillian Decker, CIR scientific analyst.

Cosmetic Ingredient Review

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Vantage Exhibit 1028
Page 1 of 37

Table 2. OSI of Jojoba Esters 15 and 60 when exposed to cosmetic actives (Brown et al. 1997).

Cosmetic active (%)	15 h	60 h
None	~35	~175
Tocopherols*	~80	~230
Iron oxides (10%) and tocopherols	~80	~225
Zinc oxides (10%) and tocopherols	~15	~90
Titanium dioxide (10%) and tocopherols	~135	~300
Malic acid (5%) and tocopherols	~110	~20
Salicylic acid (2%) and tocopherols	~25	~50
Salicylic acid (2%), titanium dioxide (10%), and tocopherols	~60	~180
Malic acid (5%), titanium dioxide (10%), and tocopherols	~120	~20
Arbutin (7%) and tocopherols	~50	~90
Kojic acid (1%) and tocopherols	~100	~250
Magnesium ascorbyl phosphate (3%) and tocopherols	~85	~170

* concentration of tocopherols not provided.

POR, 41; Sur-reply, 8-9; EX1028, 5;
EX2009, ¶¶ 140-141

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 33

Petitioner's Argument About OSI in *Trans* Isomers 2 Changed From Petition

Petition Compared Jojoba Esters and Jojoba Oil Partial Hydrogenates:

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

VANTAGE SPECIALTY CHEMICALS, INC.,

Petitioner

v.

CARGILL, INC.,

Patent Owner

Case No. IPR2023-00589

Patent No. 11,248,245

Petition for *Inter Partes* Review

Trans Isomers 2 discloses in Table 4 that the OSI of jojoba esters is greater than the OSI of partially hydrogenated jojoba oils exhibiting identical melting points. EX-1006, 3-4, Table 4; EX-1014, 7-8, Table 4; EX-1003 ¶158.

Sample	Melting Point °C	Iodine Value g I ₂ /100g of Sample	% Trans	Oxidative Stability Index, Hours	Index of Refraction n _D 20/D20 °C	Penetrometer, mm	Slip, Cadianno, Degrees (100%)	Slip, IPT, Degrees (100%)
Jojoba Esters 30	49	58	0	165	1.4418	26.4	18.9	23
Jojoba oil Partial Hydrogenate	49	69	11	27	1.4435	—	19.3	>25
Jojoba Esters 60	59	42	0	225	1.4403	9.3	17.9	14.1
Jojoba Oil Partial Hydrogenate	59	49	37	108	1.4408	5.2	18.1	15.4

Specifically, the “Jojoba Esters 30” and “Jojoba Esters 60” had OSIs of 165 and 225, respectively—significantly greater than the OSIs of the partially hydrogenated jojoba oils exhibiting identical melting points, which were 27 and 108 hours. EX-1006, 3-4, Table 4; EX-1014, 7-8, Table 4; EX-1003 ¶159. Thus, a POSA would have understood that he could achieve—and had a reasonable expectation of success of achieving—an OSI of the transesterified product (jojoba esters) that is greater than the OSI of a feedstock containing jojoba wax esters and hydrogenated jojoba wax esters. EX-1003 ¶159.

Petitioner's Argument About OSI in *Trans* Isomers 2 Changed From Petition

Board Correctly Found Partial Hydrogenate to be Commercial Product, not Feedstock:

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

VANTAGE SPECIALTY CHEMICALS, INC.,

Petitioner

v.

CARGILL, INC.,

Petitioner's reliance on Table 4's comparison of OSI values appears to be inapposite relative to the claimed OSI comparison. We see no teaching in *Trans* Isomers 2 that the "Jojoba oil Partial Hydrogenate[s]" of Table 4 form any part of a feedstock. Prelim. Resp. 20–21. To the contrary, these hydrogenates appear to be comparative *products* made by a different method—not interesterification. See Ex. 1006, 1–2 (describing a

ID, 33; Sur-reply, 10-12; Pet., 51;
EX2009, ¶¶ 164-166

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 35

Petitioner's Argument About OSI in *Trans* Isomers 2 Changed From Petition

Reply Compares Jojoba Esters 30 to Unknown Jojoba Oil Partial Hydrogenate Product with Lower OSI with No Expert Support:

UNITED STATES PATENT AND TRADEMARK OFFICE

TABLE 4. PHYSICAL PROPERTIES OF INTERESTERIFIED JOJOBA ESTERS IN COMPARISON WITH LIKE MELTING PARTIAL HYDROGENATES

Sample	Melting Point °C	Iodine Value g I/100g of Sample	% Trans	Oxidative Stability Index, Hours	Index of Refraction n_D^{20}	Penetrometer, Mm	Slip, Cadcamo, Degrees (1%)	Slip, IFT, Degrees (100%)
Jojoba Esters 30	49	58	0	165	1.4418	26.4	18.9	23
Jojoba oil Partial Hydrogenate	49	69	11	27	1.4435	—	19.3	>25
Jojoba Esters 60	59	42	0	225	1.4403	9.3	17.9	14.1
Jojoba Oil Partial Hydrogenate	59	49	37	108	1.4408	5.2	18.1	15.4

STILL COMMERCIAL PRODUCTS

PETITIONER'S REPLY TO PATENT OWNER'S RESPONSE

This table indisputably discloses that the transesterification of the claimed feedstock leads to an increased OSI in the transesterified product. For example, a comparison of rows 1 and 4 discloses that the transesterification of the claimed feedstock leads to an increased OSI in the Jojoba Esters 30 transesterified product.

Petitioner's Argument About OSI in *Trans* Isomers 2 Changed From Petition

Reply Compares Jojoba Esters 30 to Unknown Jojoba Oil Partial Hydrogenate Product with Lower OSI with No Expert Support:

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

VANTAGE SPECIALTY CHEMICALS, INC.,

Petitioner,

v.

CARGILL, INC.,

Patent Owner.

Case No. IPR2023-00589

U.S. Patent No. 11,248,245

PETITIONER'S REPLY TO PATENT OWNER'S RESPONSE

Specifically, Jojoba Esters 30 (OSI of 165) is produced by transesterifying the claimed feedstock (*i.e.*, jojoba wax esters and approximately 30% hydrogenated jojoba wax esters). EX-1014, 7; EX-1003, ¶143 (Dr. Rockstraw calculating 29.6-32.2% hydrogenated jojoba wax esters). While TI2 does not specifically disclose this feedstock's OSI, it does disclose an upper bound for that feedstock.

TI2 discloses an OSI of 108 for jojoba oil that has been partially hydrogenated (IV of 49) to a greater extent than the Jojoba Esters 30 feedstock (IV of 58). See EX-1014, 7; EX-1003, ¶101 (IV is inversely proportional to percent hydrogenation). Therefore, the OSI of the Jojoba Esters 30 feedstock—having a lower amount of hydrogenation and none of the OSI-boosting trans isomers generated during partial hydrogenation—must be lower than 108. See Ex-1014, 7 (partially hydrogenated jojoba oil generates trans isomers), Table 4; POR, 34 (“[A]s the percentage of saturated wax esters increases, the OSI value will increase.”); EX-2046, 47:4-9 (“[T]rans is known to be more stable than cis. So as you add more trans isomers to your blend, your OSI is going to go up.”).

Background References Do Not Disclose Claimed OSI Comparison

Xu 2 Compares Chemically and Enzymatically Interesterified Products

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increase in conversion degree. During a 12 week storage stability study at both 5 and 25 °C, the margarine made from enzymatically fully converted fat had the same physical properties as the margarine made from chemically interesterified fat. The margarines produced from enzymatically interesterified fats generally had much lower peroxide values compared to the margarine produced from chemically randomized fats stored at 25 °C (Zhang *et al.*, 2006). A similar observation was also apparent in a pure oil system (Tautorus and McCurdy, 1990). This confirms that enzymatic interesterification has advantages for the oxidative stability of the products.

Vantage Exhibit 1017
Page 1 of 39

Background References Do Not Disclose Claimed OSI Comparison

Lopez-Hernandez states basic chemistry and is silent as to OSI:

JFS C: Food Chemistry and Toxicology

Lipase-catalyzed Transesterification of Medium-chain Triacylglycerols and a Fully Hydrogenated Soybean Oil

ARNOLDO LOPEZ-HERNANDEZ, HUGO S. GARCIA, AND CHARLES G. HILL, JR.

[illegible]

Introduction

Introduction

Strawman modified by using either chemical or enzymatic methods to change the composition of the fatty acid and/or the head of the phospholipid. The author or their postdoctoral fellow(s) then used the modified lipid to study the effect of lipid composition on the function of the membrane. This modification is to prepare physical and/or chemical derivatives of the lipid, such as the use of fluorescent labels, fluorescent lipid analogs, or fluorescent lipid derivatives, to study the effect of lipid composition on the function of the membrane. This modification is to prepare physical and/or chemical derivatives of the lipid, such as the use of fluorescent labels, fluorescent lipid analogs, or fluorescent lipid derivatives, to study the effect of lipid composition on the function of the membrane. This modification is to prepare physical and/or chemical derivatives of the lipid, such as the use of fluorescent labels, fluorescent lipid analogs, or fluorescent lipid derivatives, to study the effect of lipid composition on the function of the membrane.

others studied). Enzyme-catalyzed transesterification reactions have been studied by several research groups because they provide a viable technology for producing SL, including acylglycerols containing medium-chain length fatty acid residues. The present investigation is focused on the production of structured TAG lipids that are rich in medium-chain fatty acid residues.

medium-chain fatty acid residues, medium-chain fatty acid residues, and long-chain fatty acid residues, respectively, saturated fatty acids with chain lengths from 6 to 12 carbon atoms. There are very few data on volatilities and liquidus points (at room temperature), and there are low volatilities and melting points (Lee and Akai, 1990). These small molecular size and relatively high solubility in water have important implications for the metabolism of these acids. The MCT

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Vantage Express

Conclusions

Structured lipids containing different levels of MCT were produced by lipase-catalyzed interesterification reactions. Studies involving 3 different commercial lipase preparations led to the conclusion that the rate of disappearance of SSS was highest when lipase TL IM was used as the biocatalyst. Six different families of new TAG were formed under all of the conditions tested, although the rates of formation, and relative concentrations of these families, depended on the initial weight ratios of the substrates and the particular biocatalyst used. The changes in the melting profiles of the final products corresponded directly to the observed depletion of high-melting-point TAG present in the FHSBO. The SL generated in these experiments contain only saturated fatty acid residues (as confirmed by GC). These SL can be considered as non-*trans* fats that provide a low-calorie alternative for several applications (baking, frying, and coating). The principal long-chain fatty acid component (stearic acid) is known to be less well absorbed in the human body. Moreover, the presence of medium-chain length fatty acid residues also confers to them a reduced caloric value relative to conventional fats and oils containing only long-chain fatty acids. The absence of unsaturated fatty acids residues implies that the products of the synthesis reaction have enhanced stability with regard to oxidation processes.

Vantage Has Not Shown Lipases
Were Known to Work on Non-
Triglycerides

Claim 1 of the '245 Patent

1. A process for transesterifying wax esters, the process comprising:
providing a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters, wherein the amount of hydrogenated jojoba wax esters is 20% to 50% by weight of the feedstock;
contacting the feedstock with a lipase; and
transesterifying the jojoba wax esters and the hydrogenated jojoba wax esters in the feedstock with the lipase to form a transesterified product;
wherein an oxidative stability index (OSI) of the transesterified product is greater than an OSI of the feedstock.

Specification includes a Sequence Listing.
Vantage Exhibit 1001
 Page 1 of 17

EX1001, cl. 1; EX2009, ¶ 95

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 41

Claim 1 of the '245 Patent

Patent Owner's Expert, Dr. Schultz, used correct meaning of lipase:

Page 1

Q (BY MR. KANE) Okay. And besides lipases or esterases, did you see any other enzyme in the prior art capable of transesterification reactions of wax ester?

MR. LUCAS: Objection to form.

A. No, I did not.

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SANTAGE EXHIBIT 1041
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Vantage Sets Up Strawman to Rebut PO's Arguments re Ester-Ester Exchange

The evidence shows that PO's assertion—that "enzymatic catalysts such as lipases were understood in the art at the time of the invention to be specific to triglycerides" (POR, 12; EX-2009, ¶¶64, 76)¹—is false. The evidence also teaches that a POSA would have known that lipases could have been—and actually were—used to transesterify jojoba esters.

transesterification as unexpected. Nor is that surprising given that enzyme-catalyzed transesterification of jojoba oil was a well-known technique for creating cosmetics ingredients by no later than 2008. See, e.g., EX-1028, 4 ("Isomerized Jojoba Oil...is...produced by the enzymatic intraesterification of Simmondsia Chinensis (Jojoba) Oil....").

Strawman Mischaracterizes PO's Arguments re Ester-Ester Exchanges

Contrary to any actual motivation, critical structural differences, namely the absence of a glycerol backbone in jojoba wax esters, would have counseled a POSITA against using a lipase with jojoba oil. EX-2009, ¶114. Vantage never addresses why a POSITA would have sought to modify Cummings or expected a lipase-catalyzed ester-ester exchange to work on jojoba wax esters. *Id.* This failure of proof infects Vantage's entire Petition, and precludes a finding of unpatentability. *Id.*

Fundamentally, Vantage fails to address Dr. Schultz's evidence about what was understood about lipase chemistry at the time of the invention, namely, that a POSITA would have believed that, to catalyze an *ester-ester exchange*, a lipase would have required a glycerol backbone structure and shorter chains that jojoba wax esters undisputedly lack. Vantage tries to give Dr. Schultz's arguments the back of the hand by dismissing the science as "misleading technical arguments." But tellingly, Vantage fails to provide *any* substantive rebuttal grounded in science and relevant to an ester-ester exchange.

Vantage's Prior Art Fails to Teach or Suggest the Claimed Invention

- Vantage's Ground References Either Do Not Disclose Lipases or Only Disclose Lipases Acting on Triglycerides
- Vantage Fails to Rebut Dr. Schultz's Explanation of Lipase Chemistry and the Importance of Glycerol Backbone
- Vantage's Sur-Reply References Do Not Disclose the Claimed Ester-Ester Exchange of Jojoba Wax Esters
- Only Ester-Ester Exchange Reaction Disclosed in Vantage's Sur-Reply References Is Admittedly Not Prior Art

Cummings Does Not Disclose Enzymatic Catalysts

Petitioner's Expert, Dr. Rockstraw:

David Rockstraw

1 UNITED STATES
2 BEFORE THE
3
4
5 VANTAGE SPECIAL
6
7 VS
8 CARGILL, INC.
9
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16 VIDEO
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18 DAVID
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20 Frida
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Q. Is there any discussion in Cummings about using an enzyme as the catalyst?

A. I don't believe there is, no.

Reported by:
Mary Agnes Drury, RPR, NYAR, CLR

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CARGILL 2046
Vantage v. Cargill
IPR2023-00589

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Xu's Lipase Catalysts Work with Triglycerides

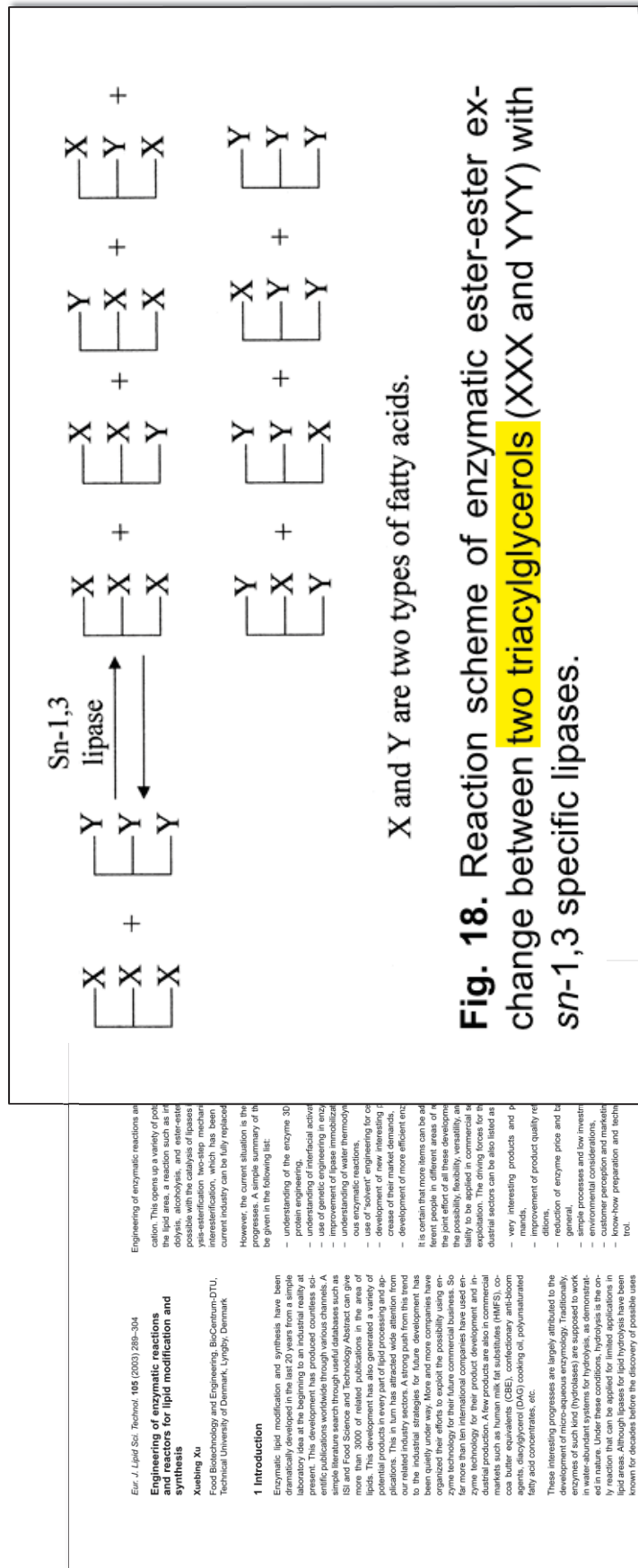


Fig. 18. Reaction scheme of enzymatic ester-ester exchange between two triacylglycerols (XXX and YYY) with sn-1,3 specific lipases.

Xu's Lipase Catalysts Work with Triglycerides

Patent Owner's Expert, Dr. Schultz:

117. I note that Xu describes in detail how lipases work on the specific triglyceride structure by exchanging *sn*-1,3 chains and maintaining the *sn*-2 chain. *Id.*, 11; *see also id.* (“The equations for the calculation of different types of **TAG** [**triacylglycerol**] species are listed below”); *id.*, 12 (“The formed **DAG** [**diacylglycerol**] can esterify with another FFA [free fatty acid] **to form a new TAG**”); *id.*, 13 (“Ester-ester exchange can also be used for the **production of structured TAGs** with the reaction between TAGs and fatty acid ethyl esters.”)

IN THE UNITED STATES OF AMERICA

In re Patent of:
U.S. Patent No.:

Issue Date:
Appl. Serial No.:
Filing Date:
Title:

I, _____, do hereby declare that the foregoing analysis may contain supplemental information

I hereby declare that all statements made by me in this declaration are true and correct to the best of my knowledge and belief, and that I am not aware of any statements and the under Section 1001

Dated: 12-6-20

1
CARGILL 2009
Vantage v. Cargill
IPR2023-00589

Xu's Lipase Catalysts Work with Triglycerides

Patent Owner's Expert, Dr. Schultz:

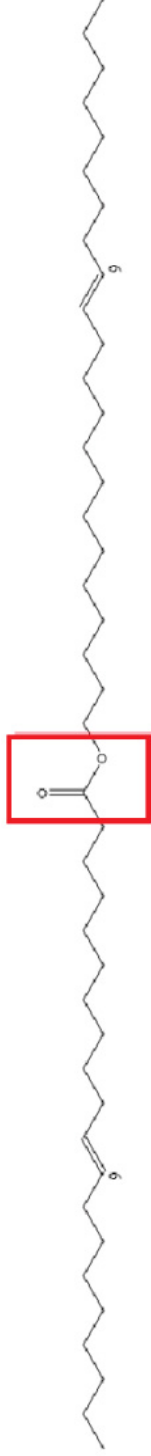
(emphasis added). In my opinion, a POSITA reading Xu would not find any explanation of how a lipase meant for use on a triglyceride could or would have been able to similarly act in an ester-ester exchange on a different structure lacking a glycerol backbone, such as jojoba wax esters. As I describe above, a POSITA would have understood that lipases have high specificity in acting on triglyceride substrates. Ex. 2039, Abstract; Ex. 2028, 6. Nothing in Xu suggests that lipases could work on substrates like jojoba wax esters, which not only lack a triglyceride, but *any* form of a glycerol.

IN THE UNITED STATES OF AMERICA,

I, _____,
In re Patent of:
U.S. Patent No.: _____
Issue Date: _____
Appl. Serial No.: _____
Filing Date: _____
Title: _____

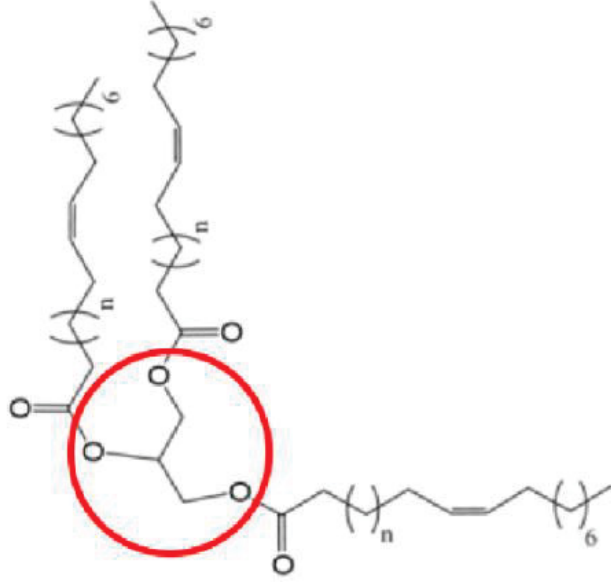
I currently have knowledge of the prior art in the field of the invention, and my analysis may conflict with the analysis of the prior art in the supplemental information. I hereby declare that all statements made in this affidavit are true and correct, and I declare that these statements and the statements and the statements under Section 1001 of the Patent Act, 35 U.S.C. 1001, are true and correct.
Dated: 12-6-20

Triglycerides and Jojoba Wax Esters Have Critical Differences

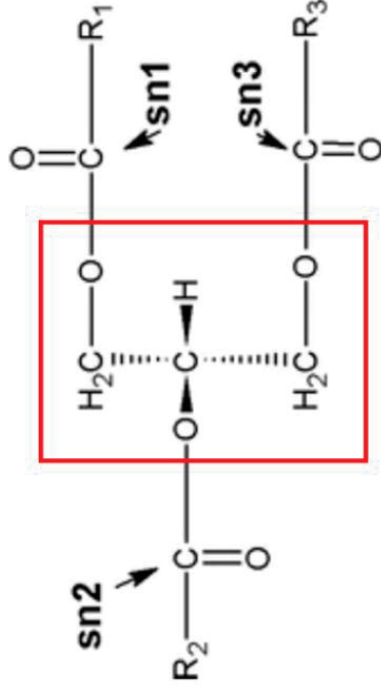


Jojoba Wax Ester Structure.

Triglycerides:

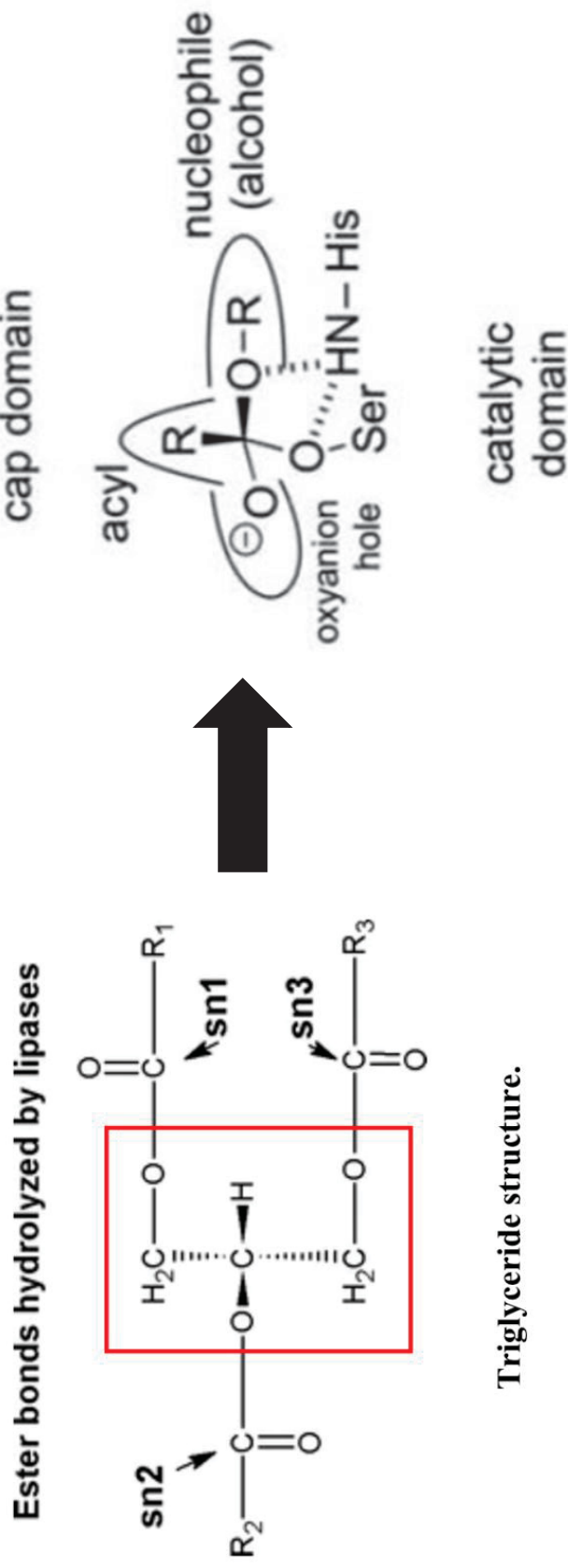


Ester bonds hydrolyzed by lipases

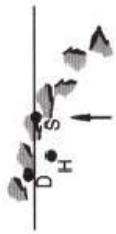
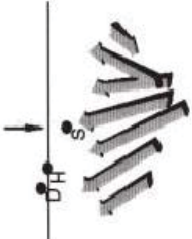
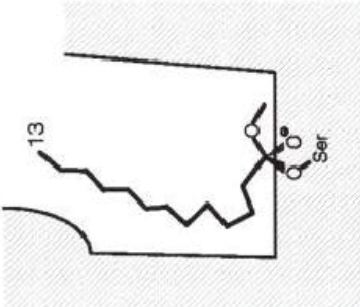
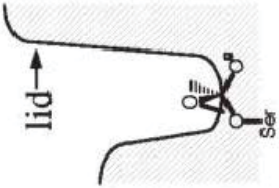
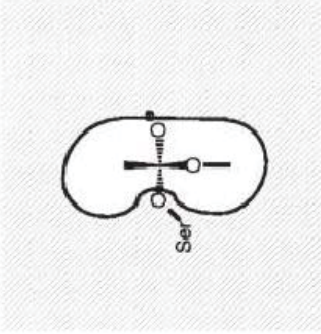
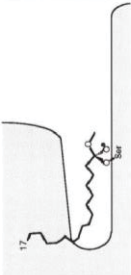
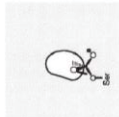
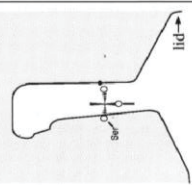


Triglyceride structure.

Lipase Catalysts Were Known to Bind with Triglycerides



Lipase Catalysts Were Known to Bind with Triglycerides

Views			
a	Side view	Front view	Top view
Views			
<i>Candida antarctica</i> lipase B			
e			
<i>Candida rugosa</i> lipase			
f			

Lipase Catalysts Were Known to Bind with Triglycerides

Patent Owner's Expert, Dr. Schultz:

84. Specifically, the structural differences from triglycerides would be expected to affect a jojoba wax ester's ability to fit into the core catalytic domain of the lipase. As I described above, lipases are typically highly specific as to the substrates they efficiently catalyze; this specificity is dictated by the size, shape, and other characteristics of the triglyceride binding site of the particular lipase. Ex. 2063, Abstract. It would not be expected that a jojoba wax ester could fit within the restricted space of the enzyme's reaction site due to the steric hindrance resulting from its large alcohol chain and its acid chain extending in the opposite direction from the ester group. Ex. 2063, Fig. 1.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020IP1
Issue Date: February 15, 2022
Appl. Serial No.: 14/578,075
Filing Date: December 19, 2014
Title: PROCESS AND SYSTEMS FOR MANUFACTURE OF DERIVATIVES

DECLARATION OF THOMAS

I currently hold the opinions set expressed in this analysis may continue, and I may acquire additional supplemental insights that may result in added observations.

I hereby declare that all statements made of information and belief that all statements made on information and belief that these statements were made with the statements and the like so made are punishable by perjury under Section 1001 of the Title 18 of the United States Code.

Dated: 12-6-23 By: Thomas

CARGILL 2009
Vantage v. Cargill
IPR2023-00589

1

FISH. POR, 29-31; EX2009, ¶ 84

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 53

Lipase Catalysts Were Known to Bind with Triglycerides

Patent Owner's Expert, Dr. Schultz:

86. Lastly, the expected interactions between the lipase's catalytic machinery and jojoba wax esters are not comparable to that of triglycerides. As discussed above, the lipase's reaction mechanism requires, *inter alia*, the catalytic triad (Ser-His-Asp), which forms the oxyanion hole when the substrate binds. Ex. 2035, 2. Unlike a triglyceride, which has three oxygens within the glycerol head to bind the catalytic triad, jojoba wax esters only have one available oxygen on the alcohol side of the ester (see figure above). This would mean that a jojoba wax ester with only this single point of contact with the catalytic triad, instead of three as in a triglyceride, would result in the jojoba wax ester being less capable of coordinating with the oxyanion hole. *Id.* A person of ordinary skill in the art would have expected that having less stability within the catalytic site would render the jojoba wax ester incapable of being transesterified with the lipase.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020IP1
Issue Date: February 15, 2022
Appl. Serial No.: 14/578,075
Filing Date: December 19, 2014
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Dated: 12-6-23 By: Thomas

Lipase Catalysts Were Known to Bind with Triglycerides

Patent Owner's Expert, Dr. Schultz:

120. As I explained in detail above, jojoba wax esters have a long alcohol and a long acid chain extending to either side of a single ester group, as opposed to the three fatty acid chains coupled through three ester groups to a glycerol backbone in a triglyceride. In my opinion, these conformational and dimensional differences are the key to why it was so unexpected for a lipase to interact with and, indeed, act upon, jojoba wax esters. See Section V(B)-(D). Unlike jojoba wax esters, the triglyceride's alcohol moiety is significantly smaller and has three separate -OH groups that can each be involved in an ester-ester exchange. Such structural differences would have been understood by a POSITA to hinder the jojoba wax ester's ability to move or position themselves to bind with the catalytic domain of the lipase binding sites. Ex. 2035, 2-3. A POSITA simply would not have expected a lipase to be able to bind to jojoba wax ester's single ester bond.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of: Jeff Addy
U.S. Patent No.: 11,248,245
47604-0020IP1
Issue Date: February 15, 2022
Appl. Serial No.: 14/578,075
Filing Date: December 19, 2014
Title: PROCESS AND SYSTEMS FOR MANUFACTURE OF DERIVATIVES

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I hereby declare that all statements made of information and belief that these statements were made with the statements and the like so made are punishable by perjury under Section 1001 of the Title 18 of the United States Code.

Dated: 12-6-23 By: Thomas

Lipase Catalysts Were Known to Bind with Triglycerides

Patent Owner's Expert, Dr. Schultz:

applicable to jojoba wax esters. In my opinion, a POSITA would have been aware of the specificity of lipase binding sites based on the size and shape of those binding sites, and would have further been aware of the differences in structures between triglycerides and jojoba wax esters. Ex. 2063, Fig. 1, 8-10. Furthermore, it is my opinion that it would not have been obvious to a POSITA that the lipase binding sites that were known to attach specifically to the glycerol backbone (i.e., the catalytic triad and oxyanion hole) at the start of an ester-ester exchange would work when exposed to molecules *lacking* that glycerol backbone. I note that Vantage

Dr. Rockstraw's Knowledge of Triglycerides and Lipases Was Limited

Petitioner's Expert, Dr. Rockstraw:

Q. Do you know if jojoba oil has sn-1 at three positions?

A. I don't understand the question.

Q. Well, you see the lipase is identified as an sn-1,3?

A. I do.

Q. Do you know -- does jojoba have that type of structure?

A. No. As I noted, I don't recall what the sn-1,3 represents in the name of the lipase.

David Rockstraw, Ph.D., P.E. -

UNITED STATES PATENT AND TRADEMARK
OFFICE
BEFORE THE PATENT TRIAL AND APPEALS

VANTAGE SPECIAL CHEMICALS, INC.,
PETITIONER,

vs
CARGILL, INC.,

PATENT OWNER.

VIDEOTAPED HYBRID DEPOSITION
of

DAVID ROCKSTRAW, Ph.D., P.E.
New York, New York
Friday, November 17, 2023

Reported by:
Mary Agnes Drury, RPR, NIAOR, GLR

Gregory Edwards, LLC | World
GregoryEdwards.com | 844

1

Dr. Rockstraw's Knowledge of Triglycerides and Lipases Was Limited

Petitioner's Expert, Dr. Rockstraw:

David Rockstraw, Ph.D., P.E. – November 17, 2023

1 UNITED STATES PATENT AND TRADEMARK OFFICE
2 BEFORE THE PATENT TRIAL AND APPEAL BOARD
3
4
5 VANTAGE SPECIAL CHEMICALS, INC.,
6 PETITIONER,
7 vs
8 CARGILL, INC.,
9 PATENT OWNER.
10
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Case No IPR2023-
US Patent No 11,

VIDEOTAPED HYBRID DEPOSITION
of
DAVID ROCKSTRAW, Ph.D., P.E.
New York, New York
Friday, November 17, 2023

Reported by:
Mary Agnes Drury, RPR, NYAOR, GLR

Gregory Edwards, LLC | Worldwide Court Reporting
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Q. Sure. Would you agree that non-specific lipases cleave acylglycerol molecules randomly generating FFAs and glycerol, as well as mono and diglycerides as intermediates?

THE WITNESS: Well, the example you use is definitely a triglyceride, because you start by talking about cleaving from the glycerol, which isn't present in jojoba oil.

And so I don't know, I haven't studied triglyceride reactions with regard to this matter.

Dr. Rockstraw's Knowledge of Triglycerides and Lipases Was Limited

Petitioner's Expert, Dr. Rockstraw:

David Rockstraw

1 UNITED STATES
2 BEFORE THE PATENT
3 OFFICE

4 VANTAGE SPECIAL

5 vs

6 CARGILL, INC.

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Reported by:

Mary Agnes Drury

Gregory Edna

Gregory

Would you expect the lipase to work
if there is not a glycerol backbone?

THE WITNESS: Well, there is not a
glycerol backbone in jojoba oil, and it
works to transesterify those, so I guess
the answer is yes, I expect it to work.

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

Vantage's Reactions are Distinguishable:

- **Esterification** facilitates **creation** of triglycerides from fatty acids and fatty alcohols; it does not require the breaking and forming of bonds between two long carbon chains like those in jojoba wax esters.
- **Hydrolysis** uses water to break down triglycerides into diacylglycerides or monoacylglycerides, not to break and reform bonds between two long carbon chains.
- **Acidolysis** and **alcoholysis** are different types of reactions that **do not** involve two long-chain esters—but instead acids and alcohols of comparably low molecular weight that lack the steric hindrance issues associated with jojoba wax esters.
- **Intraesterification** and **Isomerization** are not ester-ester exchanges; rather, these reactions refer to an exchange within a single molecule.

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1034 ('741 Publication):

- Discloses alcoholysis reaction, not ester-ester exchange
- Uses triglycerides as a substrate
- Erroneously includes jojoba oil in extensive list of predominantly triglyceride-based oils

		United States Patent Application Publication (10) Pub. No.: US 2014/0017741 A1 (43) Pub. Date: Jan. 16, 2014	
(54) ESTERIFICATION PROCESS (75) Inventors: Per Munk Nielsen, Hillebrand (DK); Hans C. Holm, Hellerup (DK)		(30) Foreign Application Priority Data Mar. 20, 2011 (EP) 11160278.1 Apr. 8, 2011 (EP) 11161192.7	
(73) Assignee: Novozymes AS, Bagsvaerd (DK) (21) Appl. No.: 14/061,609 (22) PCT Filed: Mar. 29, 2012 (86) PCT No.: PCT/EP2012/065668 371 (a)(1), (a)(2), (a)(3) Filed: Oct. 1, 2013		Publication Classification Int. Cl. C22P 7/64 (2006.01) U.S. Cl. C22P 7/64 (2013.01) USPC 435/134	
ABSTRACT The invention relates to the utilization of fatty acid feedstocks in the production of biodiesel by the use of microbial enzymes.			

1. A process for the production of fatty acid alkyl esters (FAAE) from a fatty acid feedstock, comprising the steps of:

- providing a reaction mixture 1 comprising a fatty acid feedstock, a lower alcohol and water;
- contacting the reaction mixture 1 with a lipase;
- allowing the reaction mixture 1 to react under formation of fatty acids alkyl esters;
- separation off the water/glycerol phase from the FAAE/FFA phase of reaction mixture 1;
- distilling the FAAE/FFA phase of reaction mixture 1 to obtain distilled fatty acid feedstock comprising fatty acids alkyl esters and free fatty acids;
- providing a reaction mixture 2 that comprises the distilled fatty acid feedstock and a lower alcohol;
- contacting the reaction mixture 2 with a lipase; and
- allowing the reaction mixture 2 to react under formation of fatty acids alkyl esters;

wherein water is removed from the reaction mixture 2 during any of the steps (e), (f) and (g).

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1034 ('741 Publication):

- Discloses alcoholysis reaction, not ester-ester exchange
- Uses triglycerides as a substrate
- Erroneously includes jojoba oil in extensive list of predominantly triglyceride-based oils

		United States Patent Application Publication Nielsen et al.		(10) Pub. No.: US 2014/0017741 A1 (41) Pub. Date: Jan. 16, 2014
(54) ESTERIFICATION PROCESS		(30) Foreign Application Priority Data Mar. 20, 2011 (EP) 11160278.1 Apr. 8, 2011 (JP) 11161192.7		(75) Inventors: Per Munk Nielsen, (Illinois) (DK); Hans C. Holm, (Illinois) (DK)
(73) Assignee: Novozymes A/S, (Denmark) (DK)		(51) Int. Cl. C12P 7/64 (2006.01)		(52) U.S. Cl. C12P 7/64 (2013.01)
(21) Appl. No.: 14/004,609		(22) Filed: Mar. 28, 2012		(23) Filed: Mar. 28, 2012

[0013] The term “fatty acid feedstock” is defined herein as a fatty acid feedstock substrate comprising triglyceride. The substrate may furthermore comprise fatty acid alkyl esters, diglyceride, monoglyceride, free fatty acid or any combination thereof. Any oils and fats of vegetable or animal origin comprising fatty acids may be used as substrate for producing fatty acid alkyl esters in the process of the invention.

[0037] Process Design

[0038] In the present process for enzymatic production of biodiesel a reaction mixture 1 comprising a triglyceride feedstock, a lower alcohol (e.g., methanol or ethanol) and water in a first lipase catalysed transesterification process step forms fatty acid alkyl esters, free fatty acids and free glycerol. Furthermore, glycerol may be added to the reaction mixture 1.

Sur-reply, 22-23; Reply, 5; EX1034, [0013],
[0037]-[0038]

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 62

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1034 ('741 Publication):

- Discloses alcoholysis reaction, not ester-ester exchange
- Uses triglycerides as a substrate
- Erroneously includes jojoba oil in extensive list of predominantly triglyceride-based oils

(10) United States	
(12) Patent Application Publication	
Nielsen et al.	
(43) Pub. No.: US 2014/0017741 A1	
(45) Pub. Date: Jan. 16, 2014	
	
US 2014/0017741 A1	
(54) ESTERIFICATION PROCESS	(39) Foreign Application Priority Data
(75) Inventors: Per-Mattek Nielsen, Billerød (DK); Jana C. Holm, Gentofte (DK)	Mar. 30, 2011 (EP) 11160478.1 Apr. 3, 2011 (JP) 11160192.7
(73) Assignee: Novartis AG, Basel (CH)	Publication Classification
(21) Appl. No.: 14/004,699	Int. CL
(22) PCT Filed: Mar. 28, 2012	CPC (2013.01)
(86) PCT No.: PCT/EPO12/065608	A61K 31/00 (2006.01) A61P 27/02 (2013.01) C12P 76/09 (2013.01)
(37) Filing Date: Oct. 1, 2013	U.S.C. 480/104
(21), (4) Date:	ABSTRACT
	[0014] The group consisting of coconut oil, fish oil, grape seed oil, mustard oil, palm kernel oil, flower oil, sunflower oil, halophytes, seed oil, medicinal oils or any combination thereof, wherein the oil is used as a carrier for the active ingredient.

Sur-reply, 22-23; Reply, 5; EX1034, [0014],
see also cl. 5

HSF

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1036 ('683 Publication): • Discloses replacing fatty acid chain on a triglyceride with a short chain ester to create a modified triglyceride

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
4 October 2001 (04.10.2001)

PCT WO 01/72683 A1

(51) International Patent Classification: C12C 0076, (81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, CA, CH, CN, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, FR, GB, GR, HU, IL, IN, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LU, LV, MA, MD, ME, MG, MK, MN, MU, MV, MY, NZ, PE, PG, PH, PL, PT, RU, SC, SD, SG, SI, SK, SL, TJ, TM, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(52) Publication Language: English

(54) Priority Date: 28 March 2000 (28.03.2000) US 09/555,826

(71) Applicant: THE UNITED STATES OF AMERICA, as represented by the SECRETARY OF AGRICULTURE, Department of Agriculture, Room 5N, Washington, DC 20250-0002 (US).

(72) Inventor: COMPTON, David, L., 4207 North Knoll Ridge Road, Apt. A-3, Davis, IL 61014 (US); LASZLO, Joseph A., 330 South Hawthorn Drive, Niles, IL 61041 (US).

(74) Agent: PRINCE, Stephen A., c/o, Prusker & Callie, P.O. Box 20445, Tempe, IL 60222-0445 (US).

Published with international search report

— before the expiration of the time limit for amending the claims and to be republished in the event of rectification of the claims

For two-letter codes and other abbreviations refer to the PCT Gazette.



WO 01/72683 A1

(54) Title: NOVEL SUNSCREENS FROM VEGETABLE OIL AND PLANT FENOLIS

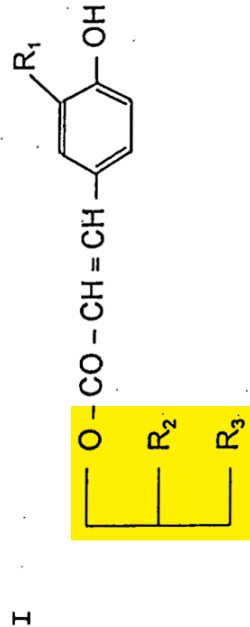
(57) Abstract: Lipase-catalyzed transesterification reactions yield novel fatty-substituted or coumaryl-substituted acylglycerols with properties suitable for use as sunscreen agents having broad spectrum UV protection. These agents have the advantage of being non-toxic, non-irritating, and non-sensitizing to the skin. They are readily incorporated into various cosmetic formulations.

VANTAGE EXHIBIT 100
Vantage v. Carpal, IPH0022-0006

Summary of the Invention

We have now discovered lipase-catalyzed reactions are useful for synthesizing ferulyl-substituted or coumaryl-substituted acylglycerols with properties suitable for use as possible sunscreen agents. These agents are readily produced in high yield by means of transesterification of a triglyceride and a ferulic or coumaric ester.

The compounds of this invention are generally characterized by Formula I:



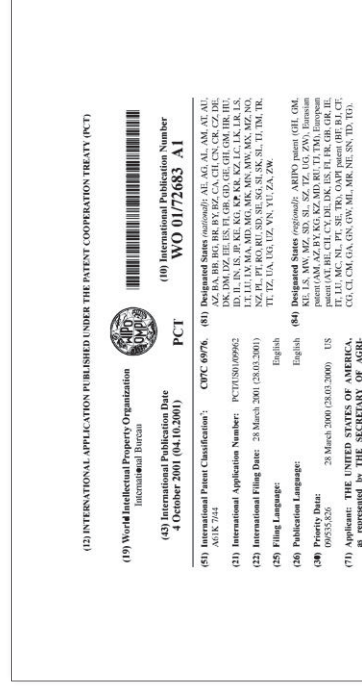
wherein $R_1 = \text{OCH}_3$ or H ; and

wherein R_2 and R_3 are each independently selected from OH and a C2-C24 fatty acid moiety.

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1036 ('683 Publication):

- Discloses replacing fatty acid chain on a triglyceride with a short chain ester to create a modified triglyceride
- **Highlights importance of glycerol backbone**
- Erroneously identifies jojoba oil as a triglyceride



Detailed Description

The substituted acylglycerols of the invention consist of a glycerol backbone in which one of the terminal hydroxyls is substituted with either a ferulate or a coumarate moiety, and at least one of the other two hydroxyls is substituted with a C2-C24 saturated or unsaturated fatty acid moiety. The natural isomers

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1036 ('683 Publication):

- Discloses replacing fatty acid chain on a triglyceride with a short chain ester to create a modified triglyceride
- Highlights importance of glycerol backbone
- Erroneously identifies jojoba oil as a triglyceride

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
4 October 2001 (04.10.2001)

PCT WO 01/72683 A1

(51) International Patent Classification: C12C 0076, A01K 7/14

(52) International Application Number: PCT/US00/09902

(53) International Filing Date: 28 March 2000 (28.03.2000)

(54) Filing Language: English

(55) Publication Language: English

(56) Priority Date: 28 March 2000 (28.03.2000) US 09/555,826

(71) Applicant: THE UNITED STATES OF AMERICA, as represented by THE SECRETARY OF AGRICULTURE

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, CA, CH, CN, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IL, IN, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LU, LV, MA, MD, ME, MG, MK, MN, MU, MV, MW, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PT, RO, RU, SD, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

(84) Designated States (international): AB, AD, AE, AF, AG, AL, AM, AN, AR, AT, AU, AZ, BA, BB, BG, BR, CA, CH, CN, CZ, DE, DK, EE, ES, FI, FR, GB, GR, GU, HK, HN, HU, ID, IL, IN, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LU, LV, MA, MD, ME, MG, MK, MN, MU, MV, MW, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PT, RO, RU, SD, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

(85) Designated States (regional): AR, AT, AU, BE, BR, CA, CH, CN, CZ, DE, DK, EE, ES, FI, FR, GB, GR, GU, HK, HN, HU, ID, IL, IN, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LU, LV, MA, MD, ME, MG, MK, MN, MU, MV, MW, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PT, RO, RU, SD, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

(86) Priority Date: 28 March 2000 (28.03.2000) US 09/555,826

(87) Applicant: THE UNITED STATES OF AMERICA, as represented by THE SECRETARY OF AGRICULTURE

19. The method of Claim 14, wherein said triglyceride is a vegetable oil selected from the group consisting of soybean oil, corn oil, sunflower seed oil, high-oleic sunflower seed oil, canola oil, safflower oil, cuphea oil, jojoba oil, coconut oil, and palm kernel oil.

WO 01/72683 A1

VANTAGE EXHIBIT 1036
Vantage v. Cargill, IP#022-00589

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1037 ('207 Publication):

- Discloses alcoholysis reaction, not ester-ester exchange
- Uses glycerides as a substrate
- Identifies jojoba oil once, in an extensive list of predominantly triglyceride-based oils



WO 2008/092207 A1

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
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(43) International Publication Date
7 August 2008 (07.08.2008)

(10) International Publication Number
PCT WO 2008/092207 A1

(51) International Patent Classification
C11C 3/00 (2006.01)
C11C 10/26 (2006.01)
C11C 10/04 (2006.01)
C11C 10/02 (2006.01)
C11C 10/00 (2006.01)
C11C 10/01 (2006.01)
C11C 10/03 (2006.01)

(21) International Application Number
PCT/US2006/000112

(22) International Filing Date: 1 February 2006 (01.02.2006)

(25) Filing Language: English

(26) Publication Language: English

(74) Agent: PHILLIPS, GUNAWARD & HETZELER, LLP
Level 21, 22A, 23, 307 Collins Street, Melbourne, Victoria
3000 (AU).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AU, AG, AL, AM, AO, AT, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CC, CD, CF, CG, CH, CI, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, IL, IN, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LI, LS, LT, LU, LV, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TD, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

Summary of the Invention

The present invention arises out of studies into processes for producing biodiesel from beef tallow. We have found that beef tallow, and other suitable animal fats and plant oils, can be treated with lipases and transesterified with ethanol or other lower alkyl alcohols to produce a biodiesel fuel that has superior properties to many currently available biodiesel fuels.

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1037 ('207 Publication):

- Discloses alcoholysis reaction, not ester-ester exchange
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(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau
(43) International Publication Date
7 August 2008 (07.08.2008)

(10) International Publication Number
PCT WO 2008/092207 A1

(51) International Patent Classification
C11C 3/00 (2006.01)
C11C 10/726 (2006.01)
C11C 10/728 (2006.01)
C11C 10/729 (2006.01)
C11C 10/730 (2006.01)
C11C 10/731 (2006.01)
C11C 10/732 (2006.01)

(41) International Application Number
PCT/AU2006/000112

(22) International Filing Date: 1 February 2006 (01.02.2006)

(25) Filing Language: Eng

(26) Publication Language: Eng

(30) Priority Date: 26/06/2005

(71) Applicant (for all designated States except US): DAL
VANTAGE FATS & OILS PTY LTD, 10/11-13, South
Road, South Australia 5207 (AU).

(72) Inventor and
Applicant (for US only): ONSCHANNIS, N
10/11-13, South Road, Pagetville 3181, South
Australia 5159 (AU).

(54) Title: BIO-DIESEL PRODUCTION

(57) Abstract: A process for producing alkyl fatty acid
treating a feedstock containing glycerides under free fatty
conditions to produce said alkyl fatty acid ester.

WO 2008/092207 A1

Glyceride containing plant oils or animal fats are particularly suitable as the feedstock

12. As used herein the term “glyceride containing plant oil or animal fat” means any oil or fat product of plant or animal origin that contains esters formed from glycerol and fatty acids (i.e. glycerides). Glycerol has three hydroxyl functional groups which can be esterified with one, two or three fatty acids to form monoglycerides, diglycerides, and triglycerides, respectively. Plant oils and animal fats contain mostly triglycerides, although they typically also contain some monoglycerides and diglycerides. The glyceride containing plant oil or animal fat may be selected from the group consisting of: animal tallow, vegetable oils, used cooking oils and fats, seeds, seed residue feedstocks, and grease trap oils.

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- Uses glycerides as a substrate
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Suitable vegetable oils that may be used in the production of biodiesel include:

rapeseed oil, soybean oil, palm oil, mustard oil, castor oil, coconut oil (copra oil), corn oil, cottonseed oil, false flax oil, hemp oil, peanut oil, radish oil, ramtil oil, rice bran oil, safflower oil, sunflower oil, tung oil, algae oil, copaiba oil, honge oil, jatrophia oil, jojoba oil, milk bush oil, petroleum nut oil, walnut oil, sunflower oil, dammar oil, linseed oil, poppyseed oil, stillingia oil, vernonia oil, amur cork tree fruit oil, apple seed oil, balanos oil, bladderpod oil, brucea javanica oil, burdock oil (bur oil), candlenut oil (kukui nut oil), carrot seed oil, chaulmoogra oil, crambe oil, cuphea oil, lemon oil, orange oil, mango oil, mowrah butter, neem oil, rosehip seed oil, sea buckthorn oil, shea butter, snowball seed oil (viburnum oil), tall oil, tamanu oil, and tonka bean oil (cumaru oil).

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Sur-reply, 23; Reply, 7; EX1037, 8:23-33

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 69

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1038 (Zoete):

- Discloses ammonolysis, not ester-ester exchange
- Reaction uses ammonia, another short chain molecule not likely to have steric hindrance problems
- Effectively an alcoholysis reaction, including with alcoholysis byproduct
- End product is an amide, not an ester

Ammonolysis of Carboxylic Esters Catalyzed by Lipases Synthesis of Fatty Acid Amides

M. C. DE ZOETE, A. C. KOCK-VAN DALEN,
F. VAN RANTWIJK, AND R. A. SHELDON
*Laboratory of Organic Chemistry and Catalysis
Delft University of Technology
2628 BL Delft, the Netherlands*

INTRODUCTION

Lipases (triacylglycerol hydrolase, EC 3.1.1.3) are stable and rugged enzymes that are used widely in a wide variety of biotechnological processes. The exploitation of their characteristics for the synthesis of a wide range of synthetic materials has been reported recently.¹ We² and others³ recently reported that ammonia acts as an unnatural acyl acceptor (ammonolysis). Carboxylic esters are converted to the corresponding carboxylic amides in essentially quantitative yield in an exothermic and, hence, irreversible reaction. The lipase from *Candida antarctica*⁴ (B type, Novozym 435) emerged from the initial investigations² as the catalyst of choice.

We wished to extend lipase-catalyzed ammonolysis² to the synthesis of fatty acid amides; these are now industrially produced from the fatty acids by a reaction with anhydrous ammonia at approximately 200 °C and 345–690 kPa and are primarily used for their lubricating and surfactant properties.⁵ A low-temperature enzymatic process would seem advantageous for particularly the thermally unstable unsaturated fatty acid amides. In the market for refined fatty acid amides, in addition to the fatty acid amides from lipids as well as the

MATERIALS Chemicals

All solvents and reagents were of reagent grade and were dried on molecular sieves before use. Novozym 435 (*Candida antarctica* B lipase on a proprietary carrier, activity of 7550 PLU/g), SP525 (a lyophilized lipase-preparation from *Candida antarctica* B), SP523 (a lyophilized lipase-preparation from *Humicola*), and Novozym IM20 (*Rhizomucor miehei* lipase on anionic resin, activity of 29 BIU/g) were kindly donated by Novo-Nordisk. Liponax, a lyophilized lipase-preparation from *Pseudomonas fluorescens* lipase, was prepared by the manufacturer according to the procedures described by the manufacturer. Novozym 435, SP525, and lipase from *P. fluorescens* were immobilized on Accusol EP100 according to a published procedure.⁶ The activities of the resulting preparations

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VANTAGE EXHIBIT 1038
Vantage v. Cargill, IPR2023-00589

We wished to extend lipase-catalyzed ammonolysis to the synthesis of fatty acid amides; these are now industrially produced from the fatty acids by a reaction with

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

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Ammonolysis of Carboxylic Esters Catalyzed by Lipases Synthesis of Fatty Acid Amides

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*Laboratory of Organic Chemistry and Catalysis
Delft University of Technology
2628 BL Delft, the Netherlands*

INTRODUCTION

Lipases (triacylglycerol hydrolases, EC 3.1.1.3) are stable and rugged enzymes that act on esters, especially on triglycerides, a wide variety of biomaterials. The exploitation of their characteristics for biocatalysis has become an essential part of the synthetic repertoire.¹ We² and others³ recently reported that ammonia acts as an unusual acyl acceptor (ammonolysis). Carboxylic esters are converted to the corresponding carboxylic amides in essentially quantitative yield in an exothermic and, hence, irreversible reaction. The lipase from *Candida antarctica*⁴ (B type, Novozym 435) emerged from the initial investigations² as the catalyst of choice.

We wished to extend lipase-catalyzed ammonolysis² as the catalyst of choice. amides; these are now industrially produced from the fatty acid and anhydrous ammonia at approximately 200 °C and 345–460 bar. The process would seem advantageous for the synthesis of particularly the thermally unstable unsaturated ones that are in demand in the market for refined fatty acid amides. In the present paper, we report on the synthesis of fatty acid amides from lipids as well as from the fatty acids.

MATERIALS AND METHODS

Chemicals and Enzymes

All solvents and reagents were of reagent grade and used without further purification. Novozym 435 (*Candida antarctica* B lipase, activity of 7550 PLU/g), SP525 (a lyophilized lipase-*pre*paration from *IM20* (*Rhizomucor miehei* lipase on anionic resin, activity 1000 PLU/g) and Novozym 435), and Novozym 435 (*Candida antarctica* B lipase, activity of 7550 PLU/g) were used according to a published procedure.⁴ The activities of the

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Vantage v. C&

Ammonolysis of Lipids

Tributyrin (10 g, 34 mmol), trilaurin (5 g, 7.8 mmol), triolein (5 g, 5.6 mmol), or jojoba wax (5 g, approximately 8.1 mmol) was dissolved in *t*-butyl alcohol (50 mL) that had been saturated with ammonia (2.5 M). Novozym 435 (250 mg) was added and the reaction mixture was shaken at 60 °C until the reaction was judged to be complete by GC (tributyrin) or TLC (other lipids). The reaction products were isolated by standard workup procedures. Full experimental details will be published elsewhere.

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1038 (Zoete):

- Discloses ammonolysis, not ester-ester exchange
- Reaction uses ammonia, another short chain molecule not likely to have steric hindrance problems
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Ammonolysis of Carboxylic Esters Catalyzed by Lipases Synthesis of Fatty Acid Amides

M. C. DE ZOETE, A. C. KOCK-VAN DALEN,
F. VAN RANTWIJK, AND R. A. SHELDON
*Laboratory of Organic Chemistry and Catalysis
Delft University of Technology
2628 BL Delft, the Netherlands*

INTRODUCTION

Lipases (triacylglycerol hydrolases, EC 3.1.1.3) are stable and rugged enzymes that are used widely in a wide variety of nonnatural reactions.¹ The hydrolytic and synthetic capabilities of these enzymes have been extensively studied, and the synthetic repertoire.¹ We² and others³ recently reported that ammonia acts as an unnatural acyl acceptor (ammonolysis). Carboxylic esters are converted to the corresponding carboxylic amides in essentially quantitative yield in an exothermic and, hence, irreversible reaction. The lipase from *Candida antarctica*⁴ (B type, Novozym 435) emerged from the initial investigations² as the catalyst of choice.

We wished to extend lipase-catalyzed ammonolysis to the synthesis of fatty acid amides; these are now industrially produced from the fatty acid anhydrous ammonia at approximately 200 °C and 345–460 kPa.⁵ A lipase-catalyzed process would seem advantageous for the synthesis of particularly the thermally unstable unsaturated ones that are in demand in the market for refined fatty acid amides. In the present paper, we report the synthesis of fatty acid amides from lipids as well as from the fatty acids.

MATERIALS AND METHODS

Chemicals and Enzymes

All solvents and reagents were of reagent grade and used without further purification. Novozym 435 (*Candida antarctica* B lipase, activity of 7550 PLU/g), SP525 (a lyophilized lipase-pretreated *Candida antarctica* B), SP523 (a lyophilized lipase-pretreated *Candida antarctica* B), IM20 (*Rhizomucor miehei* lipase on anionic resin, activity 1000 PLU/g), and Novozym 435 (a lyophilized lipase-pretreated *Candida antarctica* B) were from Novozymes A/S, Copenhagen, Denmark. SP525, SP523, and lipase from *P. doylei* were from P. doylei according to a published procedure.⁶ The activities of the

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Vantage v. Catalysts

Jojoba wax ester, which is a mixture of long-chain esters (see TABLE 1), is known to resist chemical ammonolysis at the usual conditions of 200 °C and 300–600 kPa.⁸

Ammonolysis of jojoba wax by Novozym 435 resulted in a mixture of fatty acid amides and fatty alcohols after 72 h at 60 °C. Crystallization from hexane afforded the amides (mainly *cis*-11-eicosenamide) in approximately 90% yield. The alcohols, which account for almost 50% of the starting material, were isolated by short-path-length distillation as a 1:1 mixture of *cis*-11-eicosenol and *cis*-13-docosenol.

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1035 (Lei):

- Is not prior art
- Does not disclose jojoba wax esters in ester-ester exchange with other jojoba wax esters
- Discloses reaction with short chain ethyl acetate, another short chain molecule likely to not have steric hindrance problems

Eur. J. Lipid Sci. Technol. 2015, 117, 630–636



Similarly, a research paper accepted for publication prior to the '245 patent filing (published three days later and submitted by applicants during prosecution)

Vantage's Sur-Reply Art Does Not Disclose Ester-Ester Exchange in Jojoba Wax Esters

EX1035 (Lei):

- Is not prior art
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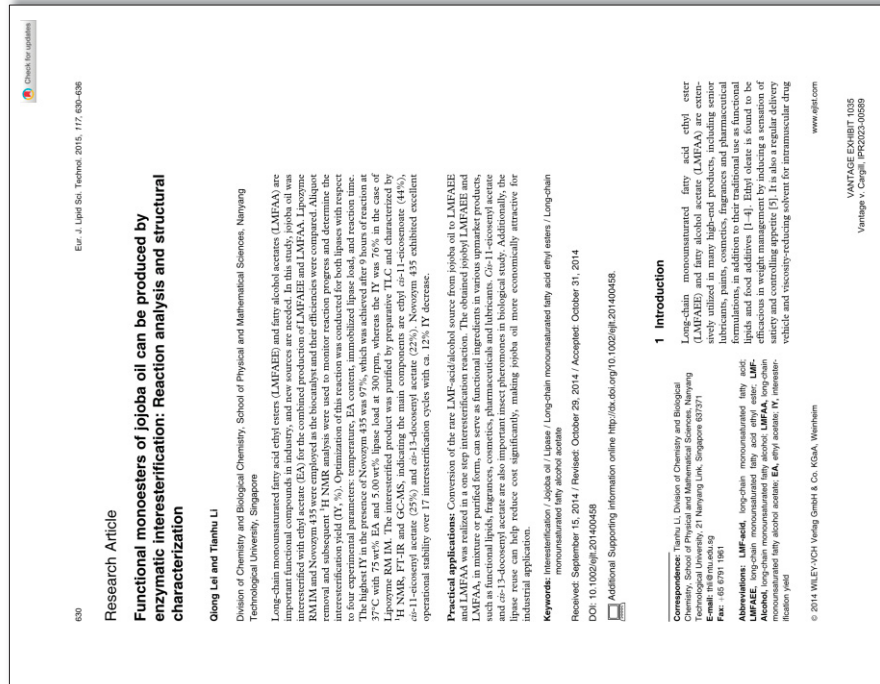
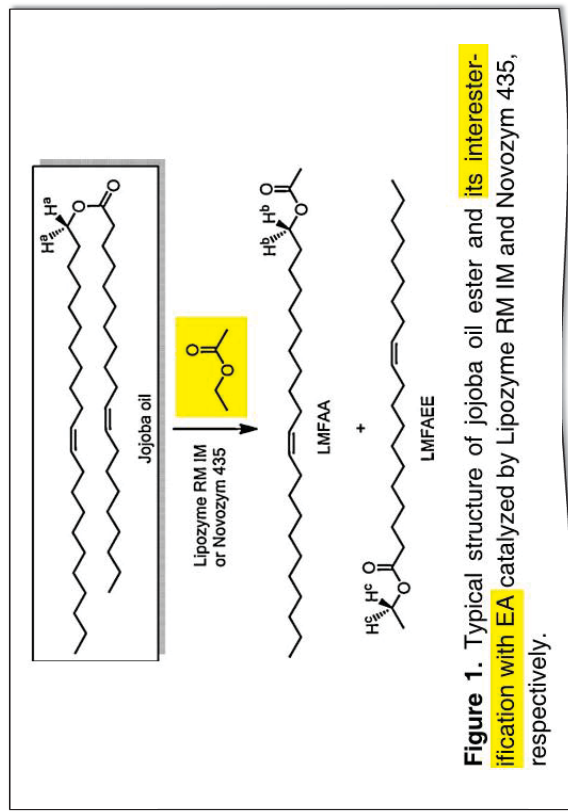
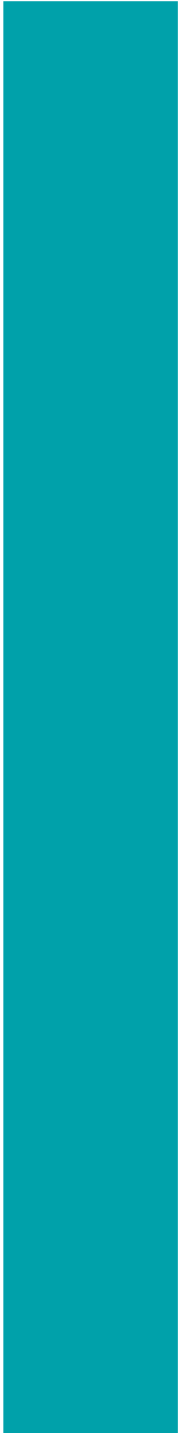


Figure 1. Typical structure of jojoba oil ester and its interesterification with EA catalyzed by Lipozyme RM IM and Novozym 435, respectively.

Sur-reply, 24-25; Reply, 5-6; EX1035, 2

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 74

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Objective Indicia Support Non-Obviousness

Objective Indicia Supports Non-Obviousness

- Prior Art and Prosecution History Show Transsterification Results Were Unpredictable
- Unpredictability Precludes Reliance on Inherency
- Vantage Tried and Failed to Make Improved Jojoba Wax Ester Product Until Finding the '245 Patent's Disclosures
- Vantage Expressed Skepticism in the '245 Patent's Method
- Vantage's Prior Art Pre-Dates Invention by Ten Years

The '245 Patent Shows Unexpected Increase in OSI After Enzymatic Transesterification

TABLE 3

Components		Feedstock Composition	Chemical Catalyst	% Change	Enzyme Catalyst	% Change
Tocopherols	Fatty Acid	0.04%	0.00%	-0.04%	0.04%	0.00%
	Methyl Esters	0.00%	3.10%	3.10%	0.00%	0.00%
	Free Fatty Alcohols	0.00%	1.80%	1.80%	0.00%	0.00%
OSI (hours)		26.5	21.5	-18.87%	38.6	45.66%
Wax Ester		98.00%	93.10%	-4.90%	98.00%	0.00%

The results also indicate that the chemically catalyzed reaction destroys the original amount of tocopherols present in the feedstock, which reduces the OSI of the transesterified product. Surprisingly, however, the OSI of the enzymatically catalyzed transesterified product is greater than that of the feedstock. This result indicates that the enzymatic catalysis has an unexpected positive effect on OSI that is not explainable simply by the enzymatic catalyst preserving the natural tocopherols originally present in the feedstock during the reaction. The OSI was measured using

Inventor Replicated Prior Art Alcoholysis Reactions with Differing Results

Gunawan's Alcoholysis Results in Increased OSI in Sunflower Oil (Experiment 1) and Decreased OSI in Jojoba Oil (Experiment 2), Showing Unpredictability:

FLORATECH002
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

DECLARATION

Applicant: Jeff Addy
Examiner: Pityu, Srikanth
Filed: December 19, 2014
Docket No.: FLORATECH002
Ser. No.: 14578075
Group Art Unit: 1657
Conf. No.: 2053

Title: Processes and Systems for Catalytic Manufacture of Wax Ester Derivatives

DECLARATION UNDER 37 C.F.R. 1.132

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir/Madam:

I, Jeff Addy, declare as follows:

1. I am a named inventor on this patent application. I am the sole inventor of the contents of this application. It is my understanding that the contents of this application are the only ones that have been maintained because the pending claims have been maintained because the obvious in view of the paper by Gunawan et al., "Alcoholysis of Plant-Based Wax-esters," *J. Oleo Sci.*, Vol. 53, No. 1, (2004) ("Gunawan") in view of Austle et al., WO2004/020099, by Bussion-Breyse et al., "Jojoba Wax: Its Esters and Components," *J. Acad. Org. Chem. Soc.*, V. 71, No. 1, (2003) ("Bussion-Breyse"), and Fucosterol (Cayman Chemicals, Inc., 2019) ("Fucosterol").

Vantage Exhibit 1002
Page 838 of 1215
Contact: 603.603.6033

	Experiment 1	Experiment 2
Reactants OSI (hrs)	5.8	24.0
Products OSI (hrs)	7.0	18.0
Δ %	20.7	-25.0

Table 4. Oxidative Stability Index of Experiments 1 & 2

Inventor Replicated Prior Art Alcoholysis Reactions with Differing Results

Patent Owner's Expert, Dr. Schultz:

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent of:	Jeff Add
U.S. Patent No.:	11,248,247 47604-0
Issue Date:	February
Appl. Serial No.:	14/578,000
Filing Date:	December
Title:	PROCESS FOR MANUFACTURING DERIVATIVE

DECLARATION

I currently hold the position of _____ at _____, a company that provides _____ services to its customers. My analysis may continue, and I will provide supplemental insights that may be relevant to the project.

I hereby declare that all statements made on this document are true and accurate to the best of my knowledge. I declare that these statements are true and accurate to the best of my knowledge, and I am not aware of any statements and the like so made by me or any other person under Section 1001 of the Title 18, Chapter 25, Subchapter I, of the Pennsylvania Statutes.

Dated: 12-6-23

CARGILL 2009
Vantage v. Cargill
IPR2023-00589

185. In my opinion, Kodali and Addy's testing support the '245 patent's disclosure of the unpredictable nature of transesterification reactions and the unexpected results achievable with the claimed invention. It was therefore surprising that the claimed invention of a lipase-catalyzed ester-ester exchange could in fact increase the OSI of the transesterified jojoba wax ester product in comparison to the feedstock.

Vantage's Prior Art Pre-Dates 2014 Invention By >10 Years

1. Cummings

Cummings, entitled “*A natural alternative*,” published in SOAP, PERFUMERY AND COSMETICS (SPC) ASIA in May 1999. EX-1004; EX-1019 ¶¶11-17. It is prior

UN
BEFORE THE PATENT TRIAL AND APPEALS BOARD
VANTAGE SPECIALTY CHEMICALS, INC.
Petitioner
v.
CARGILL, INC.
Patent Owner

2. Trans Isomers 1

Trans Isomers 1, entitled “*Trans Isomers in Cosmetics*,” published in SOAP & COSMETICS in May 2001. EX-1005; EX-1014; EX-1019 ¶¶18-25. It forms a two-

3. Trans Isomers 2

Trans Isomers 2, entitled “*Trans Isomers in Cosmetics, Part 2*,” published in SOAP & COSMETICS in June 2001. EX-1006; EX-1014; EX-1019 ¶¶18-25. It forms a

4. Brown

U.S. Patent No. RE38,141 to Brown, entitled “*Dry Emollient Compositions*,” was filed October 12, 2001, and issued June 10, 2003. EX-1007. It is prior art under

Ground 1: Claims Not Obvious Over Cummings in View of Xu and/or Sessa

Claim 1 of the '245 Patent

1. A process for transesterifying wax esters, the process comprising:
 providing a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters, wherein the amount of hydrogenated jojoba wax esters is 20% to 50% by weight of the feedstock;
 contacting the feedstock with a lipase; and
 transesterifying the jojoba wax esters and the hydrogenated jojoba wax esters in the feedstock with the lipase to form a transesterified product;
 wherein an oxidative stability index (OSI) of the transesterified product is greater than an OSI of the feedstock.

United States Patent

(12) Pat. No. 8,145,745
 (43) Pub. No. 2011/0145,745 A1
 Addy et al.

(54) PROCESS AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)

(72) Inventor: Jeff Addy, Chandler, AZ (US); Clay Addy, Chandler, AZ (US); James H. Brown, Chandler, AZ (US)

(73) Assignee: International Flora Technologies, Ltd., Chandler, AZ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 1138 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: 14/578,075

(22) Filed: Dec. 19, 2014

(65) Prior Publication Data
 US 2016/077349 A1 Jun. 23, 2016

(51) Int. Cl.
 C12P 7/64 (2006.01)

C12C 6/00 (2006.01)

(52) U.S. Cl.
 C12P 7/64 (2006.01)

(58) Field of Classification Search
 CPC: C12P 7/6458; C12C 3/005
 See application file for complete search history.

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FISH. EX1001, cl. 1; EX2009, ¶ 95

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 82

Specification includes a Sequence Listing.
 Vantage Exhibit 1001
 Page 1 of 17

Ground 1 Does Not Render Claims Unpatentable

The Combination Does Not Disclose:

1. Contacting the Jojoba Wax Ester Feedstock With a

Lipase

2. The Oxidative Stability Index (“OSI”) of the Feedstock
3. The Required Comparison of OSI Values
4. That Lipases Increase OSI of Feedstock Through
Transesterification

Ground 1 Does Not Render Claims Unpatentable

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2. The Oxidative Stability Index (“OSI”) of the Feedstock
3. The Required Comparison of OSI Values
4. That Lipases Increase OSI of Feedstock Through
Transesterification

Claim 1 of the '245 Patent

1. A process for transesterifying wax esters, the process comprising:
 providing a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters, wherein the amount of hydrogenated jojoba wax esters is 20% to 50% by weight of the feedstock;
 contacting the feedstock with a lipase; and
 transesterifying the jojoba wax esters and the hydrogenated jojoba wax esters in the feedstock with the lipase to form a transesterified product;
 wherein an oxidative stability index (OSI) of the transesterified product is greater than an OSI of the feedstock.



United States Patent
 Addy et al.

(12) Pat. No. 8,457,405
 (43) Dec. 19, 2014

(50) PROCESSES AND SYSTEMS FOR CATALYTIC MANUFACTURE OF WAX ESTER DERIVATIVES

(71) Applicant: International Flora Technologies, Ltd., Chandler, AZ (US)

(72) Inventor: Jeff Addy, Chandler, AZ (US); Clay Addy, Chandler, AZ (US); James H. Brown, Chandler, AZ (US)

(73) Assignee: International Flora Technologies, Ltd., Chandler, AZ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 118 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: 14/578,075
 (22) Filed: Dec. 19, 2014

(65) Prior Publication Data
 US 2016/077349 A1 Jun. 23, 2016

(51) Int. Cl.
 C12P 7/64 (2006.01)
 C12C 8/00 (2006.01)
 C12C 11/00 (2006.01)
 U.S. Cl.
 C12P 7/64 (2013.01); C12C 8/00 (2013.01)
 C12C 11/00 (2013.01)

(58) Field of Classification Search
 CPC C12P 7/6458; C12C 3/005
 CPC C12P 7/6458; C12C 3/005
 See application file for complete search history.

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Primary Examiner: [Name]
 Attorney: [Name]

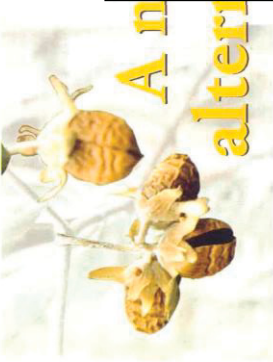
(74) Attorney: [Name]

(57) Processes for transesterifying wax esters and hydrogenated wax esters to form a transesterified product may include contacting the wax esters and hydrogenated wax esters with a lipase to form a transesterified product. The transesterified product may have an oxidative stability index (OSI) greater than the OSI of the feedstock.

Specification includes a Sequence Listing.
 Vantage Exhibit 1001
 Page 1 of 17

Cummings Does Not Disclose OSI of Feedstock

Petitioner Relies on OSI of Refined Jojoba Oil Only:



A natural alternative

Jojoba esters are a new category of naturally derived, oil free emulsifiers for a wide variety of cosmetic products. Melanie Cummings, Kd of Floratech explain

Jojoba (*Euphorbia alata*) was first used by the natives of the Southwest at one time in Mexico. Serrano people shrub known only by the name of Jojoba. The Jojoba plant bears a seed for the precious oil they could use for centuries. Jojoba has very little water and the paucity of nutrients found in the soil of its native habitat. The Jojoba plant has converted these nutrients and abundant sunshine into a versatile oil with a composition not duplicated by either man or nature.

At one time, sperm whale oil was a well known, cosmetic ingredient with Jojoba oil being used by the Aztecs and the Seri of Northern Mexico referred to jojoba as *Jojoba de Mexico*. The Jojoba plant is native to California. Padre Kino was a Jesuit priest in the region who wrote about jojoba in 1741. He and others described

how the oil was utilized by the natives people to treat wounds and burns, and to facilitate childbirth and enhance general health. When all other seed oils are refrigerated, the jojoba plant bears a pungent, dark, almost black seed oil. A liquid wax ester. This liquid wax ester can be rendered practically odorless and a popular cosmetic ingredient known as Jojoba oil.

For centuries the sperm whale was harvested for its oil to satisfy the needs of the whaling industry. The U.S. government banned the import of sperm whale oil. Jojoba oil is listed as being similar in composition to sperm whale oil.

oils & fats

Oxidative stability of many cosmetic ingredients can be measured to determine an Oxidative Stability Index (OSI).⁽¹⁾⁽¹⁰⁾ Jojoba esters demonstrate remarkable stability with OSI values of all jojoba esters greater than 100 hours and as high as 675 hours. The OSI of refined jojoba oil is around 35 hours.

Figure 3 - Jojoba ester differences

Product	Melting point °C (°F)	Index value ASTM D-154	Visual appearance	Color ASTM D-154
Jojoba esters (15)	10 - 15	79 - 85	Clear colorless liquid	< 1.5 Red
Jojoba esters (20)	32 - 48	64 - 70	White, creamy paste	< 1.0 Red
Jojoba esters (25)	47 - 51	52 - 61	White, soft paste	< 1.0 Red
Jojoba esters (30)	56 - 70	4 - 8	White crystalline powder	< 1.0 Red

Board Agreed Cummings Does Not Disclose OSI of Feedstock

On the current record, we tend to agree with Patent Owner on this point. Elsewhere, Petitioner argues that Cummings’s “refined jojoba oil” forms only “part of” the feedstock. Pet. 38. We take that to mean the *unsaturated* part of the feedstock. And, if that is so, Petitioner does not identify clearly where Cummings discloses an OSI value of the fully saturated jojoba oil that forms the *other* part of the feedstock, much less the OSI of a feedstock mixture containing both the saturated and unsaturated parts. In other words, the OSI value of “around 35 hours” that Petitioner relies upon does not appear to reflect the OSI value of the actual feedstock mixture as a whole. See Ex. 1004, 2.

Trails@uspto
571-272-7822

UNIT

BEF

Before SHER
MICHAEL A
MAJORS, AL

Tocopherols Increase OSI in Commercial Products

Final Report of the Cosmetic Ingredient Review Expert Panel

Safety Assessment of Simmondsia Chinensis (Jojoba) Seed Oil, Simmondsia Chinensis (Jojoba) Seed Wax, Hydrogenated Jojoba Oil, Hydrolyzed Jojoba Esters, Isomerized Jojoba Oil, Jojoba Esters, Simmondsia Chinensis (Jojoba) Butter, Jojoba Alcohol, and Synthetic Jojoba Oil

September 23, 2008

The 2008 Cosmetic Ingredient Review Expert Panel members are: Chairman, Wilma F. Bergfeld, M.D., F.A.C.P.; Donald V. Beltrio, M.D.; Curtis D. Klaassen, Ph.D.; Janet G. Marks, Jr., M.D.; Ronald C. Shank, Ph.D.; Thomas J. Slaga, Ph.D.; and Paul W. Snyder, D.V.M., Ph.D. The CIR Director is F. Alan Andersen, Ph.D. This report was prepared by Lillian Decker, CIR scientific analyst.

Cosmetic Ingredient Review

1101 F Street, NW, Suite 412 • Washington, DC 20004-4121 • Tel: 202/331-0800 • www.cir-safety.org

Vantage Exhibit 1028
Page 1 of 37

Table 2. OSI of Jojoba Esters 15 and 60 when exposed to cosmetic actives (Brown et al. 1997).

Cosmetic active (%)	15 h	60 h
None	~35	~175
Tocopherols*	~80	~230
Iron oxides (10%) and tocopherols	~80	~225
Zinc oxides (10%) and tocopherols	~15	~90
Titanium dioxide (10%) and tocopherols	~135	~300
Malic acid (5%) and tocopherols	~110	~20
Salicylic acid (2%) and tocopherols	~25	~50
Salicylic acid (2%), titanium dioxide (10%), and tocopherols	~60	~180
Malic acid (5%), titanium dioxide (10%), and tocopherols	~120	~20
Arbutin (7%) and tocopherols	~50	~90
Kojic acid (1%) and tocopherols	~100	~250
Magnesium ascorbyl phosphate (3%) and tocopherols	~85	~170

* concentration of tocopherols not provided.

POR, 33-34; Sur-reply, 4-7; EX1028, 7; EX2009, ¶¶ 140-141

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 88

Tocopherols Increase OSI in Commercial Products

Jojoba Esters are proper waxes, with no triglyceride components. Jojoba Esters are a complex mixture of long chain (C35 to C46) fatty acids and fatty alcohols joined by an ester bond and do not contain any trans-unsaturation (International Jojoba Export Council 2004).

Floratech (2005a,b) reported the ester chain length and saturation of 4 Jojoba Ester products as shown in Table 6. Each was reported as being 99.95% Jojoba Esters and 0.05% tocopherol.

■ ■ ■ ■

Methods of Manufacture

Simmondsia Chinensis (Jojoba) Seed Oil is combined with Hydrogenated Jojoba Oil and sodium methylate (catalyst) to get mixed Jojoba Esters (Floratech 2005a). Refined Simmondsia Chinensis (Jojoba) Seed Oil is combined with sodium methoxide to get randomized Jojoba Esters. Tocopherol is then added to make the commercial Jojoba Ester 15 (Floratech 2005b).

Final Report of the
Cosmetic Ingredient Review
Expert Panel

Safety Assessment of Simmondsia
Oil, Simmondsia Chinensis
Hydrogenated Jojoba Oil,
Isomerized Jojoba Oil, Jojoba
(Jojoba) Butter, Jojoba Alcohol

The 2008 Cosmetic Ingredient Review Expert Panel members
Donald V. Belotto, M.D., Curtis D. Klaassen, Ph.D., James
Shiga, Ph.D., and Paul W. Snyder, D.V.M., Ph.D. The CIR
prepared by Lillian Decker, CIR scientific analyst.

Cosmetic Ingredient
1101 17th Street, NW, Suite 412, Washington, DC 20036-5902

POR, 33-34; Sur-reply, 4-7; EX1028, 7; EX2009, ¶¶ 140-141

FISH

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE 89

Tocopherols Increase OSI in Commercial Products



A natural alternative

its & fats

Jojoba esters are a new category of naturally derived, oil free emollients that offer good properties for a wide variety of cosmetic products. Melanie Cummings, Kelley Dwyer and Robert Kliman of Floratex explain

Jojoba (*Simmondsia chinensis*) was first used by the Shoshone Indians at one time in western Oregon. Jojoba shrub known only by the Shoshone Indians that harvested the seeds for the precious oil they could use for many purposes. Jojoba has very little water and the paucity of nutrients found in the soil of its native habitat have resulted in a plant that converted these nutrients and abundant sunshine into a versatile oil with a unique composition. This oil is a monomeric ester, not duplicated by either any other plant or animal source.

At one time, sperm whale oil was a well known cosmetic ingredient with a reputation for its oil to satisfy the needs of the skin. For centuries the sperm whale was harvested for its oil to satisfy the needs of the skin. For centuries the sperm whale was harvested for its oil to satisfy the needs of the skin. For centuries the sperm whale was harvested for its oil to satisfy the needs of the skin.

These jojoba esters have demonstrated functional and structural properties of a broad array of personal care products. They are stable in the presence of light, heat, and oxygenated systems where emolency and oxidative stability is sometimes lacking due to the presence of unsaturated fatty acids.

Following the archetype of jojoba created by nature, jojoba esters are a new category of naturally derived, oil free emollients that offer good properties for a wide variety of cosmetic products. They are stable in the presence of light, heat, and oxygenated systems where emolency and oxidative stability is sometimes lacking due to the presence of unsaturated fatty acids.

Unique composition

While jojoba oil has unique and beneficial cosmetic properties its utility has been broadened significantly by using it as the base material for the creation of a new family of jojoba esters. **Floraesters (INCI name: jojoba esters) are unique compositions of a broad range of saturated, partially saturated and unsaturated wax esters.** Jojoba esters are made from 100% jojoba and were designed to vary in consistency from liquid to solid at ambient temperature. In the *CTFA Ingredient Handbook*, jojoba esters fall under the definition of a true ester and are not categorised within glyceryl esters, fats or oil families.

POR, 33-34; Sur-reply, 4-7; EX1004, 1; EX2009, ¶¶ 140-141

FISH

Ground 1 Does Not Render Claims Unpatentable

The Combination Does Not Disclose:

1. Contacting the Jojoba Wax Ester Feedstock With a Lipase
2. The Oxidative Stability Index (“OSI”) of the Feedstock
3. The Required Comparison of OSI Values
4. That Lipases Increase OSI of Feedstock Through Transesterification

Petitioner's Iodine Value Calculations Relate to Commercial Products

Patent Owner's Expert, Dr. Schultz:

152. Vantage fails to establish what the actual compositions of the jojoba esters “products” are and therefore cannot correlate the products’ iodine value to its feedstock. The iodine values reported by Cummings are associated with commercial jojoba ester products following transesterification. See Ex. 1004, Fig. 3. Back-calculation of the weight percentages of the original feedstock components (i.e., the reactants) from this value mistakenly relies on multiple assumptions about the products and the compounds that might form the feedstocks from which the products are formed.

Vantage v. Cargill
IPR2023-00589

Petitioner's Iodine Value Calculations Relate to Commercial Products

Sessa Uses Already Transesterified Products; Does Not Disclose Feedstock:

J Sci Food Agric 1996; 72, 295–298

Derivation of a Cocoa Jojoba Transesterified Scanning Calorimetry

David J Sessa

Biopolymer Research Unit, National Centre
Illinois 61604, USA
(Received 7 November 1995; accepted 3 June 1996)

Abstract: A series of wax-cocoa blends were prepared by transesterification of native jojoba oil with 50–500 g kg⁻¹ saturated ester. The blends were characterized by scanning calorimetry (DSC) and the resulting thermograms were compared with those of native jojoba wax and cocoa butter. The results demonstrated that the melting and crystallization behavior of the blends was a function of the amount of saturated ester. The blends were also characterized by X-ray diffraction and the results were compared with those of native jojoba wax and cocoa butter. The results showed that the blends were amorphous and the melting and crystallization behavior was a function of the amount of saturated ester. The blends were also characterized by X-ray diffraction and the results were compared with those of native jojoba wax and cocoa butter. The results showed that the blends were amorphous and the melting and crystallization behavior was a function of the amount of saturated ester.

INTRODUCTION

Differential scanning calorimetry (DSC) thermograms can give valuable information on the melting and crystallization behavior of a material. This method has been used

* This article is a US Government work and, as such, is in the public domain in the USA.
† Names are necessary to report factually on available information; however, USDA neither recommends nor endorses any particular product or brand of the product, and the use of the name by USDA implies no approval of the product or brand. The use of the name by the US Department of Agriculture is offered on a non-exclusive basis and does not constitute a warranty, nor does it imply any endorsement, national origin, religious, sex, age, marital status or handicap.

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J Sci Food Agric 0022-5142/96/\$09.00 © 1996 SCI. Printed in Great Britain

MATERIALS AND METHODS

Natural cocoa butter and the calibration sets consisting of native jojoba wax esters that were transesterified with proportionate blends of completely hydrogenated wax esters to give a series of 50, 100, 150, 200, 300, 400 and 500 g kg⁻¹ saturated ester were provided by International Flora Technologies Ltd (Gilbert, AZ, USA).

Our current research was to devise a crystallization index to estimate the level of saturation of similar blends and then to use both the fusion (Sessa *et al* 1996) and crystallization indices to select a jojoba saturation level that would give a desired level of saturation of cocoa butter.

Vantage Exhibit 1009
Page 1 of 4

Ground 2: Claims Not Obvious Over *Trans* Isomers 2 in View of Xu and/or Sessa

Ground 2 Does Not Render Claims Unpatentable

- Like Cummings, *Trans Isomers 2* does not disclose lipases or their ability to increase OSI of a partially hydrogenated feedstock
- Like Cummings, *Trans Isomers 2* relies on commercial Floraesters products that have undergone post-processing significantly affecting OSI
- Iodine values suffer same problem as Cummings

Ground 3: Claims Not Obvious Over *Trans* Isomers 2 in View of *Trans* Isomers 1 and/or Sessa

Ground 3 Does Not Render Claims Unpatentable

- Like Xu, *Trans Isomers* 1 discloses using lipases on triglycerides, not jojoba wax esters
- *Trans Isomers* 1 does not disclose OSI, therefore does not cure deficiency of *Trans Isomers* 2
- Iodine values suffer same problem as Ground 2

Trans Isomers 1 Does Not Motivate Using Lipases on Jojoba Wax Esters

Trans Isomers 1 contains a single reference to enzyme catalysts, specifically referencing triglycerides:

ALTERNATIVES TO TRANS ISOMERS IN COSMETIC PRODUCTS

The challenge of cosmetic ingredient suppliers is therefore to provide manufacturers with oxidatively stable and affordable lipid based emollients that exhibit a broad range of melting points yet which contain no trans fatty isomers. Oil phase emollients with these desirable characteristics can be obtained without the formation of trans isomers through a process known as "interesterification." Rozenaal [8] discussed use of this interesterification process to modify the melting characteristics of triglycerides using various catalysts including enzymes. Another

TRANS ISOMERS INHIBIT PROSTAGLANDIN SYNTHESIS

In vivo and in vitro studies have shown that some monounsaturated fatty acids inhibit both cyclooxygenase and lipoxygenase activities. In particular, Δ^6 -linolenic acid (Δ^6 -LNA) and Δ^5 -linolenic acid (Δ^5 -LNA) have been shown to inhibit both cyclooxygenase and lipoxygenase activities in human skin cells. In contrast, Δ^7 -linolenic acid (Δ^7 -LNA) and Δ^8 -linolenic acid (Δ^8 -LNA) have been shown to stimulate both cyclooxygenase and lipoxygenase activities. The mechanism of action of these fatty acids is not clear, but it is likely that they act through the inhibition of the cyclooxygenase and lipoxygenase enzymes. The cyclooxygenase enzyme is responsible for the conversion of arachidonic acid to prostaglandins, while the lipoxygenase enzyme is responsible for the conversion of arachidonic acid to hydroxyacids. The inhibition of these enzymes by Δ^6 -LNA and Δ^5 -LNA suggests that these fatty acids may have anti-inflammatory properties. The stimulation of these enzymes by Δ^7 -LNA and Δ^8 -LNA suggests that these fatty acids may have pro-inflammatory properties. The results of these studies suggest that the composition of the diet may play a role in the regulation of inflammation.

Trans Isomers 1 Does Not Motivate Using Lipases on Jojoba Wax Esters

Petitioner's Expert, Dr. Rockstraw, Found But Did Not Cite Rozenaal:

David Rockstraw, Ph.D., P.E. – November 17, 2023

1 UNITED STATES PATENT AND TRADEMARK OFFICE
2 BEFORE THE PATENT TRIAL AND APPEAL BOARD
3
4
5 VANTAGE SPECIAL CHEMICALS, INC.,
6 PETITIONER,
7 vs
8 CARGILL, INC.,
9 PATENT OWNER.
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Case No IPR2023-03
US Patent No 11,24

VIDEOTAPED HYBRID DEPOSITION
of
DAVID ROCKSTRAW, Ph.D., P.E.
New York, New York
Friday, November 17, 2023

Reported by:
Mary Agnes Drury, RPR, NYAOR, GLR

GregoryEdwards, LLC | Worldwide Court Reporting
GregoryEdwards.com | 844-483-2643

Q. Did you look for that Rozenaal reference?

A. I did.

Q. Did you find it?

A. I did find it.

Q. Did you cite it on in your report?

A. I did not rely on it, so I did not cite it.

Q. Why didn't you rely on it?

A. Because the content that I needed was in Trans 1?

Trans Isomers 1 Does Not Motivate Using Lipases on Jojoba Wax Esters

Like Xu, Rozenaal Discloses Triglycerides:

Chemical background

Interesterification will lead to the so-called random distribution of fatty acids over the triacylglycerols if the process is carried out in a single phase with the usual types of catalysts. A random dis-

tribution with enzymes (2). Enzymes, such as lipases, can accomplish the exchange of fatty acids at the external positions of the triacylglycerol molecule but leave the fatty acid composition at the 2-position unchanged.

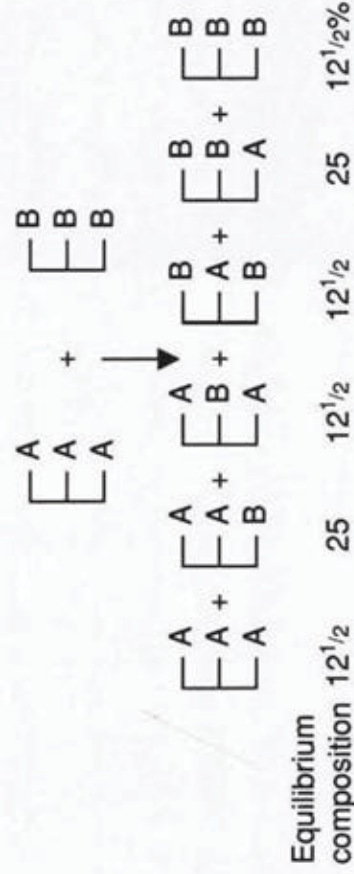


Figure 2. Interesterification of a binary mixture (50/50) of triacylglycerols

POR, 52-54; EX2030, 3; EX2009, ¶ 171

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE

FISH

Ground 4: Claims Not Obvious Over Brown in View of Xu and Cummings and/or Sessa

Ground 4 Does Not Render Claims Unpatentable

- Like Cummings and Trans Isomers 2, Brown is silent as to lipases
- Brown does not disclose OSI values
- Brown discloses alcoholysis reaction, not ester-ester exchange
- Iodine values suffer same problem as Grounds 1-3

Brown Discloses Alcoholysis Reaction, Not Ester-Ester Exchange

United States Reissued Patent Brown

(19) (12) (10) Patent N
(65) Date of F

(54) DRY EMOLLIENT COMPOSITIONS
(75) Inventor: James B. Brown, Gilbert, AZ (US)
(73) Assignee: International Flavors Technologies Ltd.,
Gilbert, AZ (US)

(*) Notice: This patent is subject to a terminal dis-
claimant.

(21) Appl. No.: 09/975,349
(22) Filed: Oct. 12, 2001
Related U.S. Patent Documents
(64) Patent No.: 6,280,246
Filed: Aug. 28, 2001
(65) Continuation-in-part of application No. 08/653,132, filed on
Oct. 12, 1997, now Pat. No. 5,986,530.

U.S. Application:
(53) Continuation-in-part of application No. 08/653,132, filed on
Oct. 12, 1997, now Pat. No. 5,986,530.

Int. Cl.
(51) U.S. Cl. 424.04, 424.02, 514.844, 514.873, 514.558
(52) U.S. Cl. 424.04, 502, 514.844, 573, 558
(58) Field of Search 424.04, 502, 514.844, 573, 558
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617,985 * 1/994 A61K2/033
62,100,000 * 1/987 A61K7/00
90,151,27 * 12/990 C11C3/10
98,262,030 * 10/997 A61K7/006



USP09753491E

The compositions comprising fatty alcohols, isopropyl esters and jojoba wax esters (jojoba oil) may be obtained by the base catalyzed alcoholysis reaction between jojoba oil and an alkyl alcohol. In the alcoholysis reaction, examples of the base catalyst materials include, but are not limited to metal alkoxides and especially alkali metal alkoxides, inorganic hydroxides, especially alkali metal hydroxides, and the like such as NaOCH₃ sodium methoxide, NaOCH₂CH₃ sodium ethoxide (potassium, calcium and lithium counterparts), KOH & NaOH (e.g., anhydrous alkali metal hydroxides in alcohol solution, with the alcohol of the solution being the alcohol used in the reaction).

Vantage Exhibit 1007
Page 1 of 13