

**VANTAGE SPECIALTY CHEMICALS, INC.,
Petitioner,
v.
CARGILL, INC.,
Patent Owner.**

**Case No. IPR2023-00589
U.S. Patent No. 11,248,245
June 11, 2024**

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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Grounds One and Two of Unpatentability

Trans Isomers 2 (Ex. 1006/1014)
or Cummings (Ex. 1004)

[with or without Sessa (Ex. 1009)]

+

Xu (Ex. 1008)

Trans Isomers 2 Teaches Chemical Transesterification

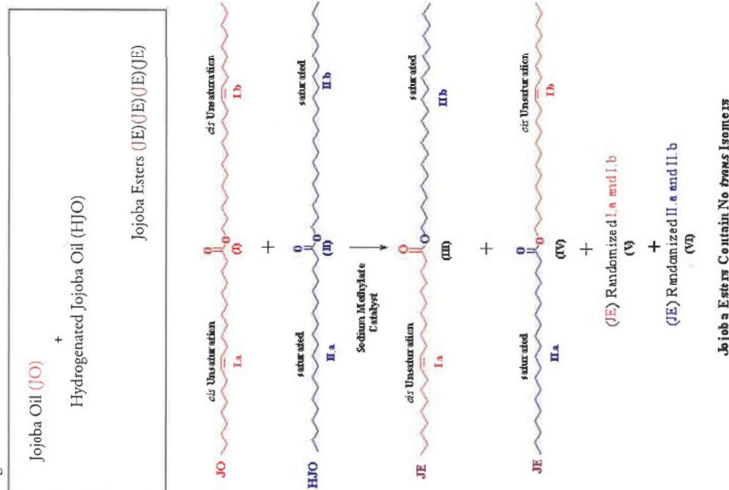


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Vantage 1048
Vantage v. Cargill

Jojoba Esters are an ideal model to use to demonstrate the *trans* free interesterification reaction and the properties of the resulting components. The CTFA Ingredient Dictionary defines Jojoba Esters as: A complex mixture of esters produced by the transesterification/interesterification of Simmondsia Chinensis (Jojoba) Oil, Hydrogenated Jojoba Oil, or a transesterified mixture of the two. Figure 4 represents the interesterification reaction between (I) jojoba oil (only *cis* isomers) and (II) fully hydrogenated jojoba oil (no *cis* or *trans* isomers).

Figure 4



Ex. 1006, p.1; Ex. 1014, p.5

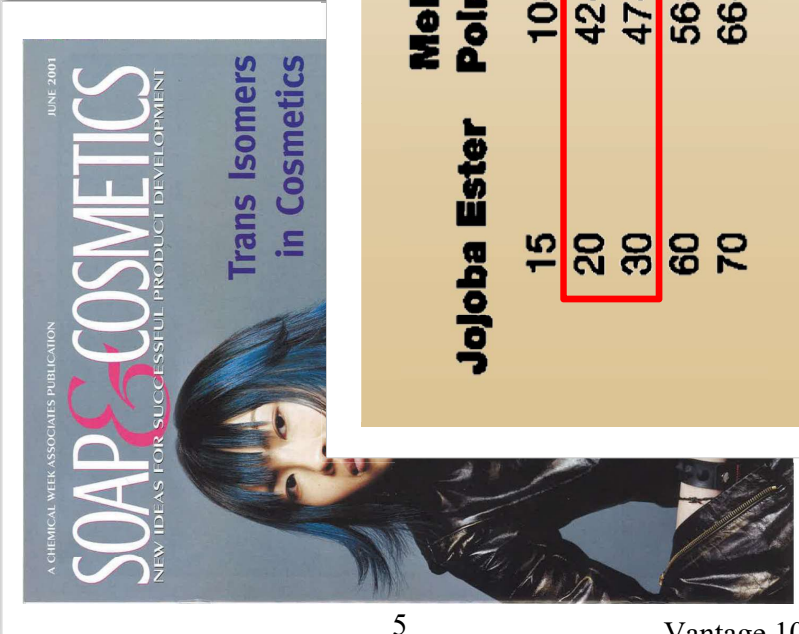
Ex. 1006, p.2; Ex. 1014, p.6

Petition, pp. 15-18

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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Trans Isomers 2 Teaches the Claimed Feedstocks



$$wt \%_{HJO} = 100 \times \frac{(IV_{JE} - IV_{JO})}{(IV_{JO} - IV_{HJO})}$$

Ex. 1003, ¶140 (annotated)

UNITED STATES PATENT AND TRADE
 BEFORE THE PATENT TRIAL AND APPEAL
 VANTAGE SPECIALTY CHEMICALS, INC.
 Petitioner,
 v.
 CARGILL, INC.

Jojoba Ester	Melting Point °C	Iodine Value g I ₂ /100g	Oxidative Stability Value, Hours	Est. Hydrogenated Jojoba Oil Content Of Feedstock (Wt %)
15	10-15	78-85	70	3.7-4.6
20	42-48	64-70	130	21-21.8
30	47-51	57-61	165	29.6-32.2
60	56-61	40-44	225	50.6-51.7
70	66-70	0-2	680	100

Ex. 1006, p.3; Ex. 1014, p.7 (annotated) Ex. 1003, ¶143

FLORATECH002

20% hydrogenated jojoba oil and 80% refined jojoba oil (by weight)

Ex. 1002, Pt. 5, p.1075

SORBSU, B92 by PQ Corporation of Valley Forge, PA along with 2.5% by weight of bleaching clay marketed under the trademark PERFORM6000 by OI-Dri Corporation of America of Chicago, IL. The treated product was then placed under vacuum for 30 minutes then subsequently filtered and cooled under nitrogen in sealed glass jars.

Results:

5. The input feedstock and the products of both experiments were analyzed for the physical properties of acid value (AV), peroxide value (PV), dropping point (DP), color measurement using Lovibond (Lovt), OSI, and iodine value (IV). The Lovt testing was done using a Lovibond PF5880 instrument. Additional testing was done to determine the comparative wax-ester carbon chain length distribution of the two products. The specific tests used to calculate each physical property for both products identified in Table 3 and are all developed by the American Oil Chemist's Society (AOCS). Table 1 lists the physical property results:

Type	AV	PV	DP	Lovt	OSI	IV
Feedstock	0.5	0.4	N/A	1.08, 3.4V	33.8	66.3
Enzymatic	0.19	0.9	48	0.98, 2.9V	117	65.7
Chemical	0.04	0.2	47.3	0.3R, 1.3V	38	66.3

Table 1 - Physical Properties

Ester	Enzymatic	Chemical
C36.0	0.10	0.11
C36.1	0.51	0.48
C36.2	0.23	0.16
C38.0	0.25	0.33
C38.1	1.89	1.74
C38.2	3.58	3.20

Vantage Exhibit 1002
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Case 1:20-cv-00001

Trans Isomers 2 Teaches Increased OSI



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Reply, pp.14-16

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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 80°C	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

Ex. 1006, p.4;
Ex. 1014, p.7
(annotated)

Sample	Melting Point °C	Iodine Value g I/100g of Sample	% Trans	Oxidative Stability Index, Hours
Jojoba Esters 30	49	58	0	165
Jojoba Oil Partial Hydrogenate	59	49	37	108



DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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Petition, pp.29-35; Reply, pp.19-21

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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

oils & fats

A natural alternative

Jojoba esters are a new category of naturally derived for a wide variety of cosmetic products. Melanie Cu of Floratech explain

[illegible]

Product	Melting point °C	Isoline cube AUCS C ₁ -25	Visual appearance
Joghurt esters (15)	10-15	78-85	Clear, colourless (liquid)
Joghurt esters (20)	42-48	64-70	White, creamy paste
Joghurt esters (30)	47-51	57-61	White, soft, paste
Joghurt ester (60)	55-61	4.0-44	White, firm paste
Joghurt esters (70)	56-70	<2	White crystalline powder

Wantage Exhibit 1004
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$$\text{wt \%}_{HJO} = 100 \times \frac{(IV_{JE} - IV_{JO})}{(IV_{JO} - IV_{HJO})} -$$

Ex. 1003, ¶104 (annotated)

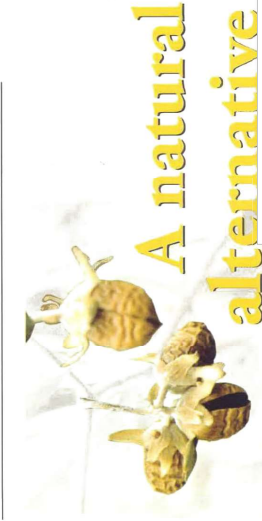
Est. Hydrogenated Jojoba Oil Content Of Feedstock (Wt %)	3.7-4.5
	21-21.3
	29.6-31.5
	50.6-50.6
	97.8-100

Ex. 1004, p.1 (annotated)

Ex. 1003, ¶107

Cummings Teaches Increased OSI

its & fats



A natural alternative

Jojoba esters are a new category of naturally derived for a wide variety of cosmetic products. Melanie C. of Floratex explain

Jojoba (*Simmondsia chinensis*) was at one time an obscure Sonoran desert shrub that grew on its foliage and the indigenous peoples that harvested its seeds for oil. For centuries, jojoba has been used as a natural skin care product. For a long time, people have known that jojoba oil is a liquid wax ester. The oil can be rendered practically anywhere and used for a variety of purposes. Jojoba oil is a popular cosmetic ingredient because of its stability and composition not duplicated by other man-made oils. In the early 1900s, the tribe of Northern Mexico referred to jojoba as *jojoba*, *jojoba de* *coahuila*, or *jojoba de* *coahuila*. The government banned the export of jojoba oil from the United States in 1914. Jojoba oil is a liquid wax ester that is composed of

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.

Ex. 1004, p.2

CARGILL, INC.

Product	Melting point °C	Iodine value g/100g
Jojoba esters (15)	10 - 15	78 - 85
Jojoba esters (20)	42 - 48	64 - 70
Jojoba Esters (30)	47 - 51	57 - 61
Jojoba Esters (50)	56 - 61	40 - 44
Jojoba esters (70)	66 - 70	< 2

Ex. 1004, p.1

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Est. Hydrogenated Jojoba Oil Content Of Feedstock (Wt %)
3.7-4.5
21-21.3
29.6-31.5
50.6-50.6
97.8-100

Ex. 1003, ¶107

>100 Hours

675 Hours

The '245 Patent Discloses No More than Cummings

(12) United States Patent Addy et al.

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

(71) Applicant: International Flavors & Fragrances, Inc., Chandler, AZ (US)

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

(73) Assignee: International Flavors & Fragrances, Inc., Chandler, AZ (US)

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

This patent is subject to a terminal disclaimer.

(21) Appl. No.: 14/578,407

(22) Filed: Dec. 19, 2014

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

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

C12C 3/00 (2006.01)

C12C 1/10 (2006.01)

(52) CPC
C12P 7/64 (2013.01); C12C 3/00 (2013.01); C12C 1/10 (2013.01)

(58) Field of Classification Search
C12P 7/64; C12C 3/00; C12C 1/10

USPC
See application file for complete search history.

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TABLE 3

Components	Feedstock Composition	Chemical Catalyst	% Change	Enzyme Catalyst	% Change
OSI (hours)	26.5	21.5	-18.87%	38.6	45.66%

Feedstock: "refined jojoba oil"

Ex. 1001, 9:27-48

Ex. 1001, 8:59

TABLE 6

Components	Feedstock Composition	Enzymatically Catalyzed	% Change
OSI (hours)	26.5	36.2	36.6%

Feedstock: "refined jojoba oil"

Ex. 1001, 11:58-12:15

Ex. 1001, 11:51-52

This result indicates that the enzymatic catalysis has an unexpected positive effect on OSI

Ex. 1001, 9:58-59

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ils & fats

A natural alternative

Jojoba esters are a new category of naturally derived for a wide variety of cosmetic products. Melanie Cu of Floratech explain

gobs (*Stromatolites*) observed at one time in the ocean. Sincere Dostei shrub known only in the mountains of the Pamirs. The indigenous people that harvested this plant for medicinal purposes used it in unique and unusual ways. The plant has efficiently converted their nutrients and abundant available water into a very little water and the purity of the water is excellent. The plant is of great medicinal value and is of great medicinal value and is of great medicinal value.

Product	Melting point °C	Index value #100g	Visual appearance
<i>Joghla</i> esters (15)	10–15	78–85	Clear, colourless liquid
<i>Joghla</i> esters (20)	42–48	64–70	White, creamy paste
<i>Joghla</i> Esters (30)	47–51	57–61	White, soft paste
<i>Joghla</i> Esters (60)	35–61	4.0–44	White, firm paste
<i>Joghla</i> esters (70)	56–70	<2	White crystalline powder

Vantage Exhibit 1004
Page 1 of 4

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.

Ex. 1004, p.2

CARGILL, INC.,

Product	Melting point °C	Iodine value g/100g
	AOCSCc 18-80	AOCSCd 1-25
Jojoba esters (15)	10 - 15	78 - 85
Jojoba esters (20)	42 - 48	64 - 70
Jojoba Esters (30)	47 - 51	57 - 61
Jojoba Esters (60)	56 - 61	40 - 44
Jojoba esters (70)	66 - 70	<2

Ex. 1004. p. 1

Est. Hydrogenated Jojoba Oil Content Of Feedstock (Wt %)	3.7-4.5
	21-21.3
	29.6-31.5
	50.6-50.6
	97.8-100

Ex. 1003. T107

Feedstock OSI ~35
JE15 OSI > 100

Petition, pp.38-41; Reply, pp.9-14

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

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Cummings' Jojoba Esters 15 and 20 Feedstocks Have the Same OSI

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

Page 140

Rockstraw: Well, I would expect a person of skill in the art would recognize that, based on the nature of the OSI device, that the OSI described here for refined jojoba oil feedstock would be similar or the same as the OSI of a feedstock that replaces part of that oil with hydrogenated oil.

oxygen or air through that sample. The oxygen in the air degrades the material, it breaks the double bonds and it creates side reactions. And the products of those side reactions are carried over with the air and captured in a second reservoir that contains water. That second reservoir has a connectivity probe in it that measures changes in connectivity. So as your sample begins to break down and you degrade those double bonds, you

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So, for instance, if I took -- the OSI device takes 5 grams of material, that's what the standard calls for you to put in it. If instead of 5 grams of refined oil, I put 4 grams of refined oil and 1 gram of fully hydrogenated wax in it, I would expect to get the same or similar result from that OSI. And in the intrinsic record I found evidence of that, so a person of skill in the art would recognize that, and it's confirmed or corroborated by the intrinsic record.

A person of skill in the art would recognize that the 5 grams of material that they put in there, if you were to replace some fraction of that with fully hydrogenated material, because that fully hydrogenated material has no -- has no effect on the stability of the oil that's there, it not like a tocopherol that's going to stabilize those double bonds, a person of skill in the art would expect to get about the same result, regardless of whether it's pure jojoba oil or some fraction of jojoba oil replaced with hydrogenated material.

Ex. 2046, 140:5-141:16, 142:21-143:7

Reply, pp.12

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DEMONSTRATIVE EXHIBIT -- NOT EVIDENCE

Dr. Rockstraw's OSI Estimate for the Jojoba Esters 20 Feedstock Is Corroborated by the Inventor's Data

FLORATECH002
enzymatic transesterification processes documented in the present specification.

Comparative Oxidative Stability Index (OSI) values for the products of both experiments and for the feedstock are included in this declaration along with various other physical properties. OSI evaluation determines the relative resistance of, for example, fats or oils to oxidation that can then be used to infer shelf life. A higher OSI value (hours) indicates a longer shelf life for a given product.

Experimental Procedure: Enzymatically Catalyzed Process

3. A 3-Neck, 1 L jacketed reaction flask equipped with an overhead agitator, vacuum, and nitrogen sparge was used as the reaction system. A total quantity of 600 g composed of 20% hydrogenated jojoba oil and 80% refined jojoba oil (by weight) was placed in the flask along with 2% w/w of the lipase enzyme marketed under the tradename LIPOZYME TL-IM by Novozymes A/S of Bagsvaerd, Denmark. The feedstock material was then mixed under vacuum at 70 C for a period of four hours. Once reaction equilibrium among chain length distribution in the forming product was observed using gas chromatography with a flame ionization detector (GC-FID), the reacted product material was filtered and cooled under nitrogen in sealed glass jars.

Experimental Procedure: Chemically Catalyzed Process

4. A 3-Neck, 1 L jacketed reaction flask equipped with an overhead agitator, vacuum, and nitrogen sparge was used as the reaction system. A total quantity of 600 g composed of 20% hydrogenated jojoba oil and 80% refined jojoba oil (by weight) was placed in the flask to form the feedstock material. A chemical transesterification process following the general industrial processes employed in the industry was then carried out. The feedstock was brought to 120 C under vacuum and dried until moisture content was <0.03% by weight. Anhydrous sodium methoxide (0.4% w/w) was added and the mixture was allowed to react under vacuum until equilibrium was detected using GC-FID. Following detection of equilibrium, 60 g of saturated brine water was then charged into the flask and mixed for 10 minutes at subtle agitation then decanted to remove sodium generated

Vantage Exhibit 1002
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CaseNo. 0000700

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A 3-Neck, 1 L jacketed reaction flask equipped with an overhead agitator, vacuum, and nitrogen sparge was used as the reaction system. A total quantity of 600 g composed of 20% hydrogenated jojoba oil and 80% refined jojoba oil (by weight) was placed in the flask along with 2% w/w of the lipase enzyme marketed under the tradename LIPOZYME TL-IM by Novozymes A/S of Bagsvaerd, Denmark.

Ex. 1002, Pt. 5, p.1075

Type	AV	PV	DP	Lovi	OSI	IV
Feedstock	0.5	0.4	N/A	1.0R 3.4Y	33.8	66.3

Ex. 1002, Pt. 5, p.1076



Reply, pp.12-13

T12 Does Not Teach the Use of Tocopherols

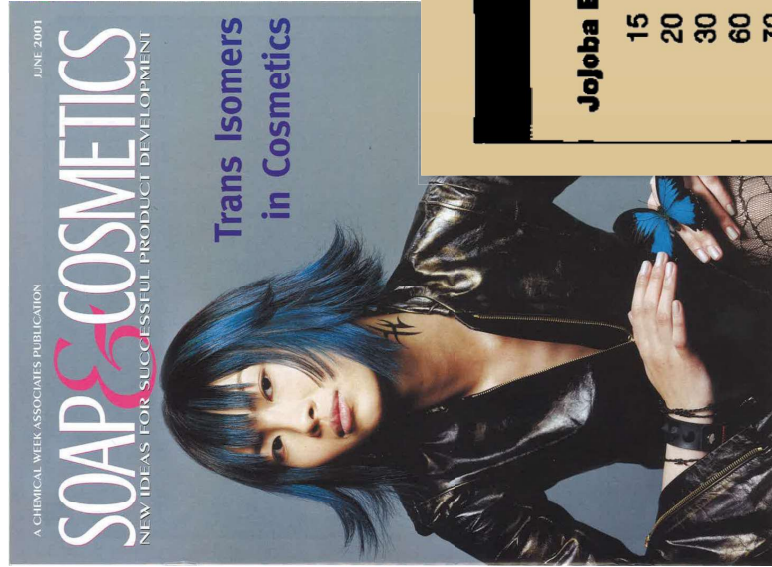


Table 2 illustrates the range of properties achievable through this interesterification process that is used to produce “Trans Free” Jojoba Esters.

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Ex. 1006, p.2; Ex. 1014, p.6

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

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Reply, pp.13-14, 16

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Ex. 1006, p.3; Ex. 1014, p.7

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Cummings Does Not Teach the Use of Tocopherols

Jojoba oil is converted to jojoba esters through a process known as interesterification. In this process, different ratios of unsaturated jojoba oil are combined with fully saturated jojoba oil and then interesterified. The process results in jojoba esters engineered to deliver specific physical, functional and chemical characteristics.

Ex. 1004, p.2

Oxidative stability

Jojoba esters are more oxidatively stable than most similar cosmetic ingredients. Typically used at levels of 5-8% in formulations, the inherent stability of jojoba esters can significantly contribute to the extended shelf life of many cosmetic preparations.⁽⁸⁾

Oxidative stability of many cosmetic ingredients can be measured to determine an Oxidative Stability Index (OSI).⁽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.

Ex. 1004, p.2

Formulation 1 - Castor Oil Free Lipstick

Ingredients	% wt/wt
A Hybrid sunflower (<i>Helianthus Annuus</i>) Oil	11.4
Macadamia ternifolia nut oil	22.0
Jojoba esters (30)	17.0
Jojoba esters (70)	1.0
Carnauba (<i>Copernicia cerifera</i>) wax	4.0
Candelilla (<i>Euphorbia cerifera</i>) wax	5.0
Beeswax	3.5
Microcrystalline wax	4.0
Polybutene	5.0
Propylparaben	qs
Silica	0.5
B Hybrid sunflower (<i>Helianthus annuus</i>) oil	13.0
Polyhydroxystearic acid	0.5
Titanium dioxide	5.5
Iron oxides	4.5
D&C Red No 30 Talc Lak ²	2.0
FD&C Blue No1	
Aluminum Lake	0.1
Iron oxides	0.5
C Tocopherol	0.1
Fragrance	qs
Total:	100.0

Ex. 1004, p.3

Vantage Exhibit 1004
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Jojoba Oil Naturally Contains Tocopherols

EX1028 also confirms that *all of* Cummings' commercial products (Jojoba

Esters 15, 20, 30, 60 and 70) contain added tocopherol:

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.

Sur-Reply, p.5

Case IPR2023-00389
U.S. Patent 11,248,245

PATENT OWNER'S SUR-REPLY
CONFIDENTIAL

CONFIDENTIAL

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

Simmondsia Chinensis (Jojoba) Seed Oil contains ~0.05% tocopherols (Yaron 1987).

Ex. 1028, p.6

Vantage Exhibit 1028
Page 1 of 37

Petition, pp.7-8; *Sur-Reply*, pp.4-6; see also Ex. 1001, 7:24-38, 8:23-25, (Table 3)

Applicant Has Represented OSI Increase Is Inherent in Lipase-Catalyzed Ester-Ester Exchange Transesterification

FILED

REMARKS

Claims 1-7 are currently pending in the application. Applicant requests reconsideration of the application in light of the following remarks.

Telephone Interview

Applicant's attorney wishes to thank the Examiner for his courtesy and time spent during the telephone interview that was held on 10/31/17.

Rejections under 35 U.S.C. 112

Claims 1-7 stand rejected by the Examiner under 35 U.S.C. 112. Specifically, in the 11/30/18 OA on pg. 6, the Examiner argues that using "wherein" in a method claim is the result of the method step rather than the method itself. The Applicant respectfully traverses this rejection. The steps of the method are recited in the first three clauses of the claim. It is apparent with the gerund form of the verbs which is required in method steps. The phase of the claim is merely describing one of the properties of the product formed. Therefore, it is unique to products made using this particular method. Therefore, it has particularly pointed out the subject matter of the claim and has included a resulting quality of the product. Applicant respectfully requests that the rejection of claims 1-7 under 112 be withdrawn.

Double Patenting Rejection

Claims 1-7 stand rejected under the judicially created doctrine of obviousness double patenting as being unpatentable over claims 8-14 of U.S. Patent Application 14/841,242 to Adeb.

The Examiner's analysis of why claims 1-7 of the present application are rejected on the claims of the '242 patent application is in error. Specifically, the Examiner p. 19 of the 11/30/17 OA that "Although the conflicting claims are not identical, they are potentially distinct from each other because the method claims in instantly presented 7 are drawn to similar compositions and similar steps as claimed in claims 8-14 of

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Vantage
Page

Rejections under 35 U.S.C. 112

Claims 1-7 stand rejected by the Examiner under 35 U.S.C. 112. Specifically, in the 11/30/18 OA on pg. 6, the Examiner argues that using "wherein" in a method claim is reciting the result of the method step rather than the method itself. The Applicant respectfully traverses this rejection. The steps of the method are recited in the first three clauses of the claim which is apparent with the gerund form of the verbs which is required in method steps. The wherein phase of the claim is merely describing one of the properties of the product formed using the method that is unique to products made using this particular method. Therefore, the Applicant has particularly pointed out the subject matter of the claim and has included a resulting quality of the product. Applicant respectfully requests that the rejection of claims 1-7 under 35 U.S.C. 112 be withdrawn.

Ex. 1002, Pt. 2, p.396

Reply, pp.16-19

Inventor Addy Admits OSI Increase Is Inherent in Lipase-Catalyzed Ester-Ester Exchange Transesterification of Jojoba

FI ORATECH002

Claims 1-7 are currently pending in the application in light of the following:

Telephone Interview

Applicant's attorney wishes to thank the Examiner for the telephone interview that was held on 10/3/17.

Rejections under 35 U.S.C. 112

Claims 1-7 and rejected by the Examiner under 35 U.S.C. 112(b) as being indefinite. The Examiner argues that the steps of the method are not clear. The steps of the method are not clear because the Examiner has not been able to determine the exact steps of the method. The Examiner has not been able to determine the exact steps of the method because the Examiner has not been able to determine the exact steps of the method. The Examiner has not been able to determine the exact steps of the method because the Examiner has not been able to determine the exact steps of the method.

Double Patenting Rejection

Claims 1-7 and rejected under the doctrine of double patenting as being unpatentable over 14/841,242 to Addy.

The Examiner's analysis of why claim 1 is unpatentable over 14/841,242 to Addy is set forth in the attached office action. Although claim 1 is unpatentable over 14/841,242 to Addy, the Examiner believes that claim 1, as originally filed reciting just wax esters in the feedstock (and not jojoba wax esters) is still fully supported and enabled by the specification as filed and reserves the right to file a claim with similar scope in a continuing application.

Vantage Exhibit 1002
Page 396 of 1215
Casefile: 10000021

EX-1002, Pt. 4, pp.948-49

Reply, pp.16-19

Inventor Addy Admits OSI Increase Is Inherent in Lipase-Catalyzed Ester-Ester Exchange Transesterification of Jojoba

CONFIDENTIAL - PURSUANT TO PROTECTIVE ORDER

UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF DELAWARE

C.A.

CARGILL INCORPORATED and
INTERNATIONAL FLORA
TECHNOLOGIES LTD.,
Plaintiffs,

vs.
VANTAGE SPECIALTY CHEMICALS INC.,
Defendant.

VIDEOTAPED 30(b) (6) DEPOSITION
OF
JEFFREY P. ADDY
Pursuant to Protective Order

DATE: August 24, 2023
TIME: 9:00 a.m.
PLACE: Fish & Richardson
3200 RBC Plaza
60 South Sixth Street
Minneapolis, Minnesota

Reported By: Christine K. Herman, RFP
Job #6040595

Verdict Legal Solutions
CONFIDENTIAL 215-241-1000 - 610-434-8388 - 302-571-0510

Q. If you do the providing, contacting and transesterifying activities that are specified in the claim prior to the wherein step, do you automatically get the OSI result in that wherein step?

A. If you do the reaction, you will get an increased oxidative stability from the feedstock.

Q.[L]et's walk through it. The providing step requires providing a feedstock comprising jojoba wax esters and hydrogenated jojoba wax esters wherein the amount of hydrogenated jojoba wax esters is 20 percent to 50 percent by weight of the feedstock. Do you see that?

A. That's what it says. Yeah.

Q. So imagine for the moment, using your experience as a chemist, doing that first step. Can you do that? Imagine it?

A. Yes.

Q. The second step says contacting the feedstock with a lipase. Can you, again, using your background, imagine contacting the feedstock we just discussed with the lipase?

A. Yes.

Q. And the transesterifying step says, transesterifying the jojoba wax esters and the hydrogenated jojoba wax esters in the feedstock with the lipase to form a transesterified product. Again, can you imagine doing that transesterifying step with the lipase of that feedstock stock?

A. I can see the reaction product to create the product, the transesterified product, yeah.

Q. And would that transesterified product automatically have a higher OSI than the feedstock's OSI?

A. The product going through the lipase reaction will have a higher OSI than the feedstock.

Ex. 1043, 60:1-61:24

Reply, pp.16-19

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DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

Sessa Teaches the Claimed Feedstock Composition

Abstract: A series of wax ester blends was constructed by transesterifying native jojoba oil with 50-500 g kg⁻¹ completely hydrogenated jojoba wax esters.

Illinois, 61604, USA

From these indices and also from regression analyses of each species' individual events we could optimise the level of saturation needed to best mimic the melt and crystallisation properties of cocoa butter. The wax ester blend with 400 g kg⁻¹ 1 saturation most closely resembled the thermal properties of cocoa butter where the monounsaturated species of the jojoba blend gave identical thermograms to tempered cocoa butter.

and crystallising temperatures as well as heats of fusion and crystallisation. This method has been used to investigate the effect of the level of saturation of jojoba wax ester blends based on heats of fusion enthalpies. The transesterification involved construction of blends of native jojoba wax ester with 50-500 g kg⁻¹ of completely hydrogenated jojoba wax ester. The objective of our current research was to devise a crystallisation index to estimate the level of saturation of similar blends and then to use both the fusion (Sessa *et al* 1996) and crystallisation indices to determine the degree of saturation of the blends that most closely mimics the thermal properties of cocoa butter.

Ex. 1009, Abstract

Table B

Hydrogenated Jojoba Wax Ester Content of Blend (g kg ⁻¹)	Wt% Hydrogenated Jojoba Wax Ester
50	5
100	10
150	15
200	20
300	30
400	40
500	50

$$\times \frac{1 \text{ kg of Blend}}{1000 \text{ g of Blend}} \times 100 \text{ wt \%}$$

Ex. 1003, ¶110

Xu Provides Strong Motivation to Use a Lipase to Catalyze Transesterification Including Ester-Ester Exchange

Eur. J. Lipid Sci. Technol. 195 (2003) 298–304
Engineering of enzymatic reactions and reactors for lipid modification and synthesis

Xuesong Xu
Food Biotechnology and Engineering, Biocentrum-DTU,
Technical University of Denmark, Lyngby, Denmark

1 Introduction

Enzymatic lipid modification and synthesis have been

Engineering of enzymatic reactions and reactors 299
cation. This opens up a variety of potential applications. In the lipid area, a reaction such as interesterification (acylolysis, alcoholysis, and ester-ester exchange) is also possible with the catalysis of lipases in a probable hydrolysis-esterification two-step mechanism. Thus chemical interesterification, which has been implemented in the current industry can be fully replaced.

However, the current situation is the joint result of many progresses. A simple summary of these progresses can be given in the following list:

This opens up a variety of potential applications. In the lipid area, a reaction such as interesterification (acylolysis, alcoholysis, and ester-ester exchange) is also possible with the catalysis of lipases in a probable hydrolysis-esterification two-step mechanism. Thus chemical interesterification, which has been implemented in the current industry can be fully replaced.

6 Enzymatic ester-ester exchange

Ester-ester exchange is a reaction between two esters. Acids will be exchanged between the hydroxyl moieties. Chemical ester-ester exchange, often referring to as chemical interesterification, is dominantly used for the randomization of oils and fats, which has been widely applied in industry for oil and fat modification. Enzymatic ester-ester exchange is an alternative to the chemical method for the modification of oils and fats.

Ex. 1008, p.289

Ex. 1008, pp.298-99

Correspondence: Xuesong Xu, Biocentrum-DTU, Technical University of Denmark, Lyngby, Denmark.
Phone: +45-452-527-73, Fax: +45-455-545-22, e-mail: xu@biocentrum.dtu.dk

Vantage Exhibit 1008
Page 1 of 16

Vantage 1048

Vantage v. Cargill

Petition, pp.23-24, 36-37, 49-50; Reply, pp.1, 8, 23

The Lipase Arguments in Patent Owner's Response

Lipases are enzymes that, prior to the '245 Patent, were known to be specific to triglycerides. EX-2009, ¶64; *see also* EX-2033, 1; EX-2034, 6; EX-2035, 10.

VANTAGE SPECIALTY CHEMICALS, INC.

Petitioner

POR, p.2

While chemical catalysts were known for use with both triglycerides and jojoba oil, enzymatic catalysts such as lipases were understood in the art at the time of the invention to be specific to triglycerides. EX-2009, ¶64; EX-2033, 1; EX-2035, 10.

PATENT OWNER'S RESPONSE

POR, p.12

Prior to the filing of the '245 Patent, no literature described any attempts to transesterify anything other than triglycerides with a lipase, let alone a structurally dissimilar compound like jojoba wax esters. EX-2009, ¶76-77.

POR, p.15

Clearly, the POSITA was not motivated over those three decades to attempt the enzymatic transesterification of jojoba wax esters and any contrary suggestion today is hindsight. *Id.*

POR, p.31

 Check for updates

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Eur. J. Lipid Sci. Technol. **108** (2003) 601–607 DOI: 10.1002/ejlt.2003006053

Geoffrey Hills

Industrial use of lipases to produce fatty acid esters

A wide range of fatty acid esters can be synthesised by esterification and transesterification reactions catalysed by lipases in non-aqueous systems. As well as modifications to the natural substrate triacylglycerol, many other commercially important substrates

Diapace AG,
Project House Biotechnology,
Hansa-Wolfgang,
Germany

Eur. J. Lipid Sci. Technol. **105** (2003) 601–607 DOI 10.1002/ejlt.200300853 601

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Confession: While

Coauthors: Lilla

Industrial use of lipases to produce fatty acid

A wide range of fatty acid esters can be synthesised by esterification and transesterification reactions catalysed by lipases in non-aqueous systems. As well as modifications to the natural substrate triglycerol, many other commercially important substrates can be produced using this enzyme technology, so lipases have been chosen for this project.

Dejussa AG,
Projekt House Biotechnology,
Hansau-Wolfgang,
Germany

Lipases are well known to catalyse hydrolysis, esterification and transesterification reactions on a wide range of substrates [1-3]. As well as hydrolysing their natural substrate triglycerides, this group of enzymes are very versatile in a variety of bioconversions on natural and synthetic substances, ranging from regio- and stereoselective hydrolyses to syntheses in organic, non-aqueous media. Many alcohols other than glycerol can be substrates for lipases, including simple n-alcohols and polyhydroxy compounds such as carbohydrates. Reactions can be carried out on the complete range of carboxylic acids from acetic acid to greater than C_{20} fatty acids.

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VANTAGE EXHIBIT 1033
Vantage v. Carmill IPR2023-00589

Ex. 1033, p.601

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 Check for updates

Eur. J. Lipid Sci. Technol. 2016, 117, 630–638

Research Article

Functional monoesters of jojoba oil can be produced by enzymatic interesterification: Reaction analysis and structural

630

37°C with 75 wt% EA and 5.00 wt% lipase load at 300 rpm, whereas the IV was 76% in the case of Lipzyme RM IM. The interesterified product was purified by preparative TLC and characterized by ¹H NMR, FT-IR and GC-MS, indicating the main components are ethyl *cis*-11-icosanoate (44%), *trans*-11-icosenoic acid (22%), *cis*-13-docosyl acetate (22%) and *trans*-13-docosyl acetate (12%). The interesterification reaction was stable over 17 interesterification cycles with ca. 12% IV decrease.

In this study, jojoba oil was interesterified with ethyl acetate (EA) for the combined production of LMFAEE and LMFAA.

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Received: September 13, 2011
DOI: 10.1002/ajb.201400459

☐ Additional Supporting Information online <http://dx.doi.org/10.1002/eilt.201400458>.

Keywords: [Interesterification](#) / [Jojoba oil](#) / [Lipase](#) / [Long-chain monounsaturated fatty acid ethyl esters](#) / [Long-chain monounsaturated fatty alcohol acetate](#)

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VANTAGE EXHIBIT 1035
Vantage v. Cardill. IPR2023-00589

Ex. 1035, p. 630

DEMONSTRATIVE EXHIBIT – NOT EVIDENCE

The Lipase Arguments in Patent Owner's Sur-Reply

Vantage ultimately fails to provide any evidence showing why a POSITA would have found it obvious to use lipases to catalyze an *ester-ester exchange* in jojoba wax esters.

First, Vantage does not substantively rebut Dr. Schultz's evidence of lipase chemistry and his testimony that "critical structural differences, namely jojoba wax esters' absence of a glycerol backbone, would have counseled a POSITA against using a lipase with jojoba wax esters."⁵ EX2009, ¶114; *see also id.*, ¶¶69-87.

Sur-Reply, p. 16

Case IPR2023-00589
U.S. Patent 11,248,245

PATENT OWNER'S SUR-REPLY
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Second, Vantage focuses on different lipase reactions and different substrates rather than the claimed *ester-ester exchange* of a mixture of long-chain jojoba wax esters. As Dr. Schultz explained, at the time of the invention lipase-catalyzed *ester-ester exchange specifically was believed to require a glycerol backbone to interact with the lipase's catalytic triad and ester chains of no more than 18 carbons* EX2009, ¶¶76-81; EX2063, 3, 10. A POSITA would have had no motivation or expectation of success in using a lipase to catalyze an ester-ester exchange on jojoba wax esters, which lack a glycerol backbone and *have two long (20-22) carbon chains attached to a single ester bond*. EX2009, ¶¶53, 82-87, 119-121. Reactions that use small molecules, such as alcohols or acids, would not give a POSITA any reasonable expectation that an ester-ester exchange of a molecule with two long carbon chains, like jojoba wax esters, would be successful.

Sur-Reply, p. 17

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The Lipase Arguments in Patent Owner's Sur-Reply

UNITED STATES PATENT AND TRADEMARK OFFICE	
BEFORE THE PATENT TRIAL AND APPEAL BOARD	
VANTAGE SPECIALTY CHEMICALS, INC. Petitioner,	
v.	
CARGILL, INCORPORATED, Patent Owner	
Case IPR2023-00589 U.S. Patent 11,248,245	
PATENT OWNER'S SUR-REPLY CONFIDENTIAL	
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A POSITA would have had no motivation or expectation of success in using a lipase to catalyze an ester-ester exchange on jojoba wax esters, which lack a glycerol backbone and have two long (20-22) carbon chains attached to a single ester bond. EX2009, ¶¶53, 82-87, 119-121.

Sur-Reply, p.17

Lipases Were Known to Catalyze Reactions on Jojoba Esters

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

M. C. DE ZOETER, A. C. KOCK-VAN DALEN,

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.

marked 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 by a one-pot procedure.

Ammonolysis of jojoba wax by Novozym 435 resulted in a mixture of fatty acid amides and fatty alcohols after 72 h at 60 °C.

Novozym 435 (a lipase preparation from *Humicola* sp.) and Novozym 435 (a lipase preparation from *Humicola* sp.) were kindly donated by Novo-Nordisk. Liponax, a lipase preparation from *Pseudomonas* sp., was kindly donated by Gist-brocades. SP525, SP523, and lipase from *P. alcaligenes* were immobilized on Accurel EP100 according to a published procedure.⁶ The activities of the resulting preparations

VANTAGE EXHIBIT 1028
Vantage v. Cargill, F02023-00589

Ex. 1038, pp.347-48

Ex. 1028, p.4

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

Isomerized Jojoba Oil (no CAS No.) is defined as a mixture of esters produced by the enzymatic intraesterification of Simmondsia Chinensis (Jojoba) Oil (q.v.). Its chemical classification is ester. It has both plant and synthetic sources

The 2008 Cosmetic Ingredient Review Expert Panel members are: Chairman, Wilma F. Bergfeld, M.D., F.A.C.P.; Donald A. Belsito, M.D.; Curtis D. Kelsch, M.D.; James G. Marks, Jr., M.D.; Ronald C. Shank, Ph.D.; Thomas J. Slomkowski, M.D.; and William H. Stein, M.D. The CIR Director is F. Alan Andersen, Ph.D. This report was prepared by Lillian Becker, CIR scientific analyst.

Cosmetic Ingredient Review

1101 17th Street, NW, Suite 412 • Washington, DC 20005-1702 • Tel: 202-331-0661 • Fax: 202-331-0688 • cirinfo@cirfoundation.org

Vantage Exhibit 1028
Page 1 of 37

Reply, pp.5-7

Lipases Were Known to Catalyze Ester-Ester Exchange Interesterification on Jojoba Esters

In this study, jojoba oil was interesterified with ethyl acetate (EA) for the combined production of LMFAEE and LMFAA. Lipozyme RM IM and Novozym 435 were employed as the biocatalyst and their efficiencies were compared.

Ex. 1035, p. 630

Three grams of jojoba oil and a certain amount of EA were added into a sealed round bottom flask. After stirring the substrates at given temperature for 5 minutes, a certain amount of Lipozyme RM IM or Novozym 435 was added to start the reaction.

Ex. 1035, p. 631

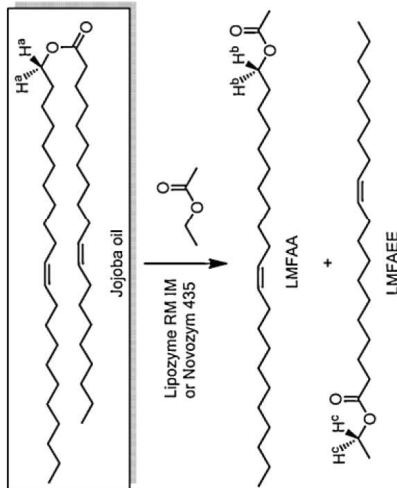


Figure 1. Typical structure of jojoba oil ester and its interesterification with EA catalyzed by Lipozyme RM IM and Novozym 435, respectively.

Ex. 1035, p. 631

Reply, pp. 5-7

DEMONSTRATIVE EXHIBIT - NOT EVIDENCE

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Cargill's Secondary Consideration Allegations

Unexpected and Surprising Results



Copying



Age of Prior art



