Natural Emollient Esters from Bio-Based Succinic Acid

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INTRODUCTION

BioAmber is a next generation chemicals company, with a portfolio of renewable chemicals; a C4 Platform, based on bio-based succinic acid and its derivatives, and a C6 Platform, which includes bio-based adipic acid and other bio-based C6 chemicals. BioAmber was first to market with commercial scale bio-based succinic acid, and is currently serving customers from a toll manufacturing plant in France. Based on growing market demand for bio-based chemicals, BioAmber is constructing a large-scale plant in Ontario, Canada, to supply industrial scale bio-based succinic acid. As a platform chemical, bio-based succinic acid has a wide range of applications, including as an intermediate for natural emollient esters.

Bio-based succinic acid is a natural raw material, which can be used produce a wide range of personal care ingredients including ester solvents, emollient esters, and surfactants. Made from plants, bio-based succinic acid can be reacted with natural alcohols to produce bio-based esters. C8-C18 esters have been shown to exhibit unique emollient properties for skin and hair care. While this paper will focus on the performance of selected emollient esters, C2-C5 succinate esters have been shown to be excellent solvents for personal care applications including fragrance carriers and nail enamel removers. Additionally, bio-based succinic acid is an effective preservative and effervescent agent.

Natural emollient esters based on BioAmber's bio-based succinic acid have been found to exhibit a range of feels depending on their molecular weights. The emollient esters are soluble in a variety of cosmetic base fluids, providing ease of formulation with these natural ingredients. The emollients have a dry-feel, and provide a silky, powdery non-greasy sensory signal. In addition, the succinate esters provide excellent shine and gloss.

BioAmber bio-based succinic acid is carbon neutral at industrial capacity. The Sarnia, Ontario plant will generate a savings of >100% greenhouse gas emissions, and uses 60.9% less energy compared to petrochemical production of adipic acid kilogram per kilogram.[1] The use of bio-based succinic acid from BioAmber immediately reduces the overall footprint of any product, when used as a direct replacement for energy and carbon intensive petrochemicals.

BioAmber bio-based succinic acid can be used to make natural succinate esters that can be formulated into Personal Care applications that are 100% natural and non-GMO. Diesters from BioAmber's bio-based succinic acid are renewable alternatives to petroleum based emollient esters for personal care. They provide high shine, excellent feel, are easily formulated, are mild and non-irritating, and can be up to 100% derived from plants when using plant based alcohols.

FORMULATIONS

Three natural succinate diesters were synthesized by reacting BioAmber's bio-based succinic acid with the corresponding alcohols (Table 1). After screening a number of diesters for feel, three succinate esters were selected in order to examine the properties they impart to skin and hair care formulations. Oleyl alcohol (unsaturated C18 straight chain alcohol) was reacted with bio-based succinic acid to form dioleyl succinate (DOS). Dioleyl succinate is a natural heavy-feel emollient ester. Stearyl alcohol (saturated C18 straight chain alcohol) was reacted with bio-based succinic acid to form distearyl succinate. Distearyl succinate is a solid diester that has potential as a heavy-feel emollient, as well as a pearlescent agent. A mixture of caprylic and capric alcohols was used to synthesize the mixed ester, octyl decyl succinate (ODS). ODS is a light-feel succinate ester that is soluble in a range of cosmetic fluid bases. All of these succinate esters are natural emollients esters, made from plants, with up to 100% bio-based carbon as defined by ASTM D6866.

Table1. Natural Succinate Emollient Esters		
Succinate ester	Succinate Ester Molar Mass (g/mol)	Physical State
Octyl Decyl succinate	371 on average	Liquid
Dioleyl succinate	619	Liquid
Distearyl succinate	623	Solid (mp 67 °C)

The succinate esters were evaluated for feel, and formulated into skin and hair care products based on their performance. The sensory evaluations of the diesters, the formulations, and the evaluation of the formulated products compared to the controls were conducted by Susan Raffey Consulting.[4]

RESULTS AND DISCUSSION

Octa Decyl Succinate

ODS was tested for its solubility in a variety of cosmetic fluids (Table 2). 10 Wt-% of ODS was combined with solvent at room temperature. While ODS was insoluble in water and butylene glycol, it was soluble in a number of other cosmetic fluids including a variety of oils, dimethicone fluid, and isopropyl myristate. The wide range of solubility allows for easy formulations of the natural succinate diester into a variety of personal care formulations.

Table2. Solubility of octyl decyl succinate[2]	
Solvent (INCI Name)	Solubility
Water	Insoluble
Butylene glycol	Insoluble
Isopropyl myristate	Soluble
Caprylic/capruic triglyceride	Soluble
Dimethicone fluid	Soluble
Ethyl trisiloxane	Soluble
Safflower oil	Soluble
Castor oil	Soluble
Mineral oil	Soluble
Hydrogenated polyisobutylene	Soluble

ODS was tested for skin irritation using the Repeat Insult Patch Test conducted at AMA Laboratories. Testing was conducted on neat ODS with a semi-occlusive patch on 50 subjects. The patch was applied directly to the skin for 24 hours, and then removed. The procedure was repeated for 9 consecutive exposures with 24 hours between each exposure for three consecutive weeks. Skin reactions are scored just before applications two through nine. After a 10-14 day rest period, a retest dose was applied to a previously unexposed area. RIPT testing showed no response for any test subject over the course of the test,[3] indicating ODS is a mild and non-irritating natural emollient ester.

Octyl decyl succinate (ODS) has a high refractive index at 1.45, which makes it an excellent natural glossing agent. On its own, ODS was found to have a substantial 'glide' feel, similar to jojoba or castor oils. In addition, the ODS has very low odor, making it an excellent candidate for non-fragranced formulations.

Due to the high shine of ODS, it was formulated into a Hair Shine Serum at 5.0 wt-%. A positive control was also formulated, which contained 5.0 wt-% of cyclopentasiloxane (Cosmetic fluid 995). The complete formulation is available as Table 2 in the Formulation Appendix. ODS was compatible with the silicone base fluid, and the hair shine serum containing the natural octyl decyl succinate was found to have equal clarity and color compared to the control. The ODS containing hair shine serum was also found to have a richer and more luxurious feel, was less greasy compared to the control, and dry evaporated to a velvety finish. Overall, the ODS was found to impart positive sensory attributes to the hair shine serum, as well as provide a bio-based natural ingredient.

Heavy Feel Emollients: Dioleyl Succinate and Distearyl Succinate

Dioleyl succinate was formulated into a hair conditioner at 5.0 wt-%. It was used as a one-for-one replacement of 350 cps dimethicone. The complete formulation is available in Table 1 of the Formulation Appendix. The hair conditioner using dioleyl succinate had better opacity compared to the control formulation. In addition, the conditioner was significantly creamier and richer. Finally, the dioleyl succinate imparted a more substantial feel during rub out.

Distearyl succinate was formulated into a moisturizing cream at 3.0 wt-%. It was used as a one-for-one replacement of dimethicone and dimethicone cross-polymer. The complete formulation is available in Table 3 of the Formulation

Appendix. The moisturizing cream with distearyl succinate was thicker than the control formulation at room temperature. The cream had excellent feel, with an average rub-out. After rub-out, the formulation with distearyl succinate had slightly lower gloss, but better creaminess. The lower gloss is most likely due to distearyl succinate being a solid succinate ester.

CONCLUSION

Bio-based succinic acid is a versatile intermediate for new natural personal care ingredients. Made from non-GMO plant feedstock, BioAmber's bio-based succinic acid can be used as a preservative, an effervescent agent, and an intermediate for natural emollient esters and solvents. Additionally, BioAmber's bio-based succinic acid is carbon neutral at industrial scale. When reacted with natural alcohols, a range of natural emollient esters can be produced. These emollient esters impart a silky, powdery light, dry, non-greasy sensory signal to hair and skin care formulations. In addition, liquid emollient esters are excellent glossing agents, imparting high shine to hair care formulations. The esters are easily formulated into oil in water emulsions, and are up to 100% derived from plants. Finally, the esters are mild and non-irritating, giving an excellent combination of performance and environmental profile.

BioAmber

BioAmber is a next generation chemicals company. Its proprietary technology platform combines industrial biotechnology, an innovative purification process and chemical catalysis to convert renewable feedstocks into chemicals for use in a wide variety of everyday products including plastics, food additives and personal care products. BioAmber produces bio-based succinic acid in what it believes to be one of the world's largest bio-based chemical manufacturing facilities. For more information visit the company's web site at www.bio-amber.com.

REFERENCES

1. "Field-to-Gate Energy and Greenhouse Gas Emissions Associated with Succinic Acid Produced At BioAmber's Facility In Sarnia Ontario," Riffel Consulting, March 2013
2. Solubility testing conducted at Cosmetech Laboratories, 39 Plymouth Street, Fairfield, NJ.
3. RIPT testing conducted at AMA Laboratories, 216 Congers Road, New City, NY. Dermatologist signed full reports available upon request.
4. Formulations and sensory evaluations conducted at Susan Raffy Consulting, 3420 W Macarthur Blvd., Santa Ana, CA

BIOGRAPHIES

Tara Mullen is the Manager of Application Engineering and Technical Support at BioAmber Inc. She received a Batchelor of Science Degree in Chemistry from St. Norbert College, and a Doctorate Degree in Polymer Science and Engineering from the University of Southern Mississippi. Prior to joining BioAmber in 2012, Tara held Application and Commercial Development roles at Segetis, and also spent 5 years at GE Plastics in a Product Development role.

Susan Raffy, president of Susan Raffy Consulting, has more than 25 years of experience in the personal care industry. After obtaining her Bachelor of Arts degree in chemistry from the University of California, San Diego, Susan's first position was developing pharmaceuticals and nutritional supplements for American McGaw. She also briefly worked as a research assistant in pulmonary medicine at the University of Texas's Health Science Center/Veterans Administration Hospital before making her foray into cosmetic formulation as associate scientist in the Allercreme Cosmetics division of Alcon Laboratories Inc. in 1985. After Alcon, Raffy continued formulating at Physicians Formula Cosmetic Inc., where she was a scientist responsible for new product development using innovative raw materials and advanced technology for hypoallergenic skin treatment. In 1990, she joined Aware Products Inc. as director of research and development/quality assurance. Her experience then transitioned to business, with positions as VP of business development/technical sales for GAR Laboratories Inc. and technical sales representative and industry specialist for Lipscomb Chemical Company Inc. Raffy went back to formulating as the director of product development for the beauty division of Guthy-Renker Corp. At the same time, she began teaching at the Fashion Institute of Design and Merchandising, where she was named Outstanding Instructor of the Year. Following, she was president of CTF Concepts Inc. and senior sales manager and technical director for Randall International LP before beginning her consulting business in 2008. Raffy is an active member Society of Cosmetic Chemists, where she has served as chair, summer event coordinator and newsletter editor for the California chapter. She is also an active member of the American Chemical Society and Beauty Industry West.

FORMULATION APPENDIX

Appendix Table 1. Hair Conditioner Formulation with Dioleyl Succinate[4]

	Trade Name	INCI Name	% w/w
Part A	Deionized water	Deionized water	82.3
	Cetearyl alcohol	Cetearyl alcohol	6.0
	Polysorbate 60	Polysorbate 60	1.0
	Lexamine S-13	Stearamidopropyl dimethylamine	1.5
	Adogen 432 CG	Dicetyldimonium chloride	3.0
	Panthenol	Panthenol	0.1
Part B	Citric acid	Citric acid	0.1
Part C	Botanistat PF-64	Phenoxyethanol, Caprylyl glycol, Ethylhexylglycerine, hexylene glycol	1.0
Part D	Dioleyl succinate*	Dioleyl succinate	5.0

*Positive control replaced Part D with 5.0% w/w of Cyclopentasiloxane (Cosmetic Fluid 995)

Appendix Table 2. Hair Shine Serum Formulation with Octyl Decyl Succinate[4]

	Trade Name	INCI Name	% w/w
Part A	Cosmetic Fluid 9109	Cyclotetrasiloxane, cyclopentasiloxane, dimethicone	94.9
	Octyl decyl succinate*	Octyl decyl succinate	5.0
	Lovespell type Fragrance	Fragrance	0.1

*Positive control replaced octyl decyl succinate with 5.0% w/w of Cyclopentasiloxane (Cosmetic Fluid 995)

Appendix Table 3. Moisturizing Skin Cream Formulation with Distearyl Succinate[4]

	Trade Name	INCI Name	% w/w
Part A	Deionized water	Deionized water	74.4
	Carbopol Ultrez 10	Carbomer	0.5
Part B	Glycerin 99.50%	Glycerine	4.0
	Keltrol CG	Xanthan Gum	0.2
Part C	Safflower Oil High Oleic	Carthamus tinctorius (Safflower) seed oil	5.0
	Isopropyl Myristate	Isopropyl myristate	2.0
	Cetyl alcohol	Cetyl alcohol	2.0
	Stearic acid	Stearic acid	3.0
	Glyceryl stearate SE	Glyceryl stearate SE	2.0
	DM 100	Dimethicone	1.0
	Shea Butter	Butyrospermum parkii (Shea) nut butter	1.0
Part D	Triethanolamine 99%	Triethanolamine	0.8
Part E	Vitamin E Acetate	Tocopheryl Acetate	0.1
Part F	Botanistat PF-64	Phenoxyethanol, Caprylyl glycol, Ethylhexylglycerine, hexylene glycol	1.0
Part G	Distearyl succinate*	Distearyl succinate	3.0

*Positive control replaced Part G with 3.0% w/w dimethicone and dimethicone cross polymer (Botanisil GB-40)