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(54) **Titre : PLASTIFIANTS D'ESTERS ALKYLIQUES D'ACIDE GRAS EPOXYDES ET PROCEDES DE FABRICATION DE PLASTIFIANTS D'ESTERS ALKYLIQUES D'ACIDE GRAS EPOXYDES**

(54) **Title: EPOXIDIZED FATTY ACID ALKYL ESTER PLASTICIZERS AND METHODS FOR MAKING EPOXIDIZED FATTY ACID ALKYL ESTER PLASTICIZERS**

(57) **Abrégé/Abstract:**
Epoxidized fatty acid alkyl esters and methods for making epoxidized fatty acid alkyl esters. Such epoxidized fatty acid alkyl esters can be prepared by epoxidizing fatty acid alkyl esters with an acid and a peroxide. Epoxidation can be performed under controlled reaction conditions to provide epoxidized fatty acid alkyl esters having an iodine value in the range of from 4 to 15 g I_2 / 100 g of epoxidized fatty acid alkyl esters. Epoxidized fatty acid alkyl esters can be employed in plasticizer compositions, either alone or in combination with other plasticizers, such as epoxidized natural oils. Such plasticizers in turn may be used in the formation of polymeric compositions.

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(54) Title: EPOXIDIZED FATTY ACID ALKYL ESTER PLASTICIZERS AND METHODS FOR MAKING EPOXIDIZED FATTY ACID ALKYL ESTER PLASTICIZERS

(57) Abstract: Epoxidized fatty acid alkyl esters and methods for making epoxidized fatty acid alkyl esters. Such epoxidized fatty acid alkyl esters can be prepared by epoxidizing fatty acid alkyl esters with an acid and a peroxide. Epoxidation can be performed under controlled reaction conditions to provide epoxidized fatty acid alkyl esters having an iodine value in the range of from 4 to 15 g I_2 / 100 g of epoxidized fatty acid alkyl esters. Epoxidized fatty acid alkyl esters can be employed in plasticizer compositions, either alone or in combination with other plasticizers, such as epoxidized natural oils. Such plasticizers in turn may be used in the formation of polymeric compositions.

EPOXIDIZED FATTY ACID ALKYL ESTER PLASTICIZERS AND METHODS FOR MAKING EPOXIDIZED FATTY ACID ALKYL ESTER PLASTICIZERS

FIELD

[0001] Various embodiments of the present invention relate to methods for making epoxidized fatty acid alkyl esters. Such epoxidized fatty acid alkyl esters may be employed as plasticizers or in plasticizer compositions.

INTRODUCTION

[0002] Plasticizers are compounds or mixtures of compounds that, when added to polymer resins, can lower one or more of the modulus and tensile strength, and increase one or more of flexibility, elongation, impact strength, and tear strength of the resin (typically a thermoplastic polymer) to which they are added. A plasticizer may also lower the melting point of the polymer resin, which lowers the glass transition temperature and enhances processability of the polymer resin.

[0003] Phthalic acid diesters (also known as "phthalates") are commonly used as plasticizers in many flexible polymer products, such as polymer products formed from polyvinyl chloride ("PVC") and other vinyl polymers. Examples of phthalate plasticizers include diisononyl phthalate, diallyl phthalate, di-2-ethylhexyl-phthalate, dioctyl phthalate, and diisodecyl phthalate. Other plasticizers used for high temperature applications are trimellitates and adipic polyesters.

[0004] Phthalate plasticizers have recently come under intense scrutiny by public interest groups concerned about the negative environmental impact of phthalates and potential adverse health effects in humans exposed to phthalates. Accordingly, plasticizers that minimize or eliminate the use of phthalates are needed. Although advancements have been made, improvements in such plasticizers are still desired.

SUMMARY

[0005] One embodiment is a plasticizer composition comprising: epoxidized fatty acid alkyl esters, wherein said epoxidized fatty acid alkyl esters have an iodine value in the range of from 4 to 15 g I_2 / 100 g of epoxidized fatty acid alkyl esters.

[0006] Another embodiment is a process for producing epoxidized fatty acid alkyl esters, said process comprising: epoxidizing fatty acid alkyl esters via controlled epoxidation by contact with an acid and an aqueous peroxide solution to form epoxidized fatty acid alkyl esters, wherein said controlled epoxidation comprises selecting a reaction temperature, a reaction time,

an aqueous peroxide solution concentration, and a peroxide solution feed rate to cause said epoxidized fatty acid alkyl esters to retain sufficient unsaturation to present an iodine value in the range of from 4 to 15 g I_2 / 100 g of epoxidized fatty acid alkyl esters.

[0006A] The present specification discloses and claims a plasticizer composition comprising: epoxidized fatty acid alkyl esters, wherein said epoxidized fatty acid alkyl esters have an iodine value in the range of from 7 to 10 g I_2 / 100 g of epoxidized fatty acid alkyl esters, wherein said epoxidized fatty acid alkyl esters have an oxirane oxygen content of at least 6 weight percent based on the entire weight of said epoxidized fatty acid alkyl esters, and wherein said epoxidized fatty acid alkyl esters are prepared from esterified soybean oil. Also disclosed and claimed is a polymeric composition comprising a polymeric resin and such a plasticizer composition.

[0006B] The present specification also discloses and claims a process for producing epoxidized fatty acid alkyl esters, said process comprising: epoxidizing fatty acid alkyl esters via controlled epoxidation by contact with an acid and an aqueous peroxide solution to form epoxidized fatty acid alkyl esters, wherein said controlled epoxidation comprises selecting a reaction temperature, a reaction time, an aqueous peroxide solution concentration, and a peroxide solution feed rate to cause said epoxidized fatty acid alkyl esters to retain sufficient unsaturation to present an iodine value in the range of from 7 to10 g I_2 / 100 g of epoxidized fatty acid alkyl esters, wherein said epoxidized fatty acid alkyl esters have an oxirane oxygen content of at least 6 weight percent based on the entire weight of said epoxidized fatty acid alkyl esters, and wherein said fatty acid alkyl esters are prepared by transesterifying a soybean oil.

DETAILED DESCRIPTION

[0007] Various embodiments of the present invention concern methods for preparing epoxidized fatty acid alkyl esters ("eFAAE") from esterified natural oils. Such eFAAEs can be employed as a plasticizer alone or in combination with an epoxidized natural oil ("eNO"). Plasticizers comprising eFAAE and optionally eNO can be employed with a variety of polymeric resins and in making various articles of manufacture.

Preparing Epoxidized Fatty Acid Alkyl Esters

[0008] The eFAAE can be prepared by epoxidation of an esterified (e.g., transesterified) natural oil. Thus, in one or more embodiments, the eFAAE can be prepared by first subjecting a natural oil to esterification (e.g., transesterification), thereby producing fatty acid alkyl esters. The term "natural oil" denotes an oil comprising fatty acid triglycerides and is derived from a microbe (algae, bacteria), a plant/vegetable, and/or a seed. In an embodiment, the natural oil includes genetically-modified natural oil. In another embodiment, the natural oil excludes petroleum-derived oil. Non-limiting examples of suitable natural oils include algae oil, beef tallow oil, canola oil, castor oil, corn oil, fish oil, linseed oil, palm oil, rapeseed oil, safflower oil, soybean oil, sunflower oil, tall oil, tung oil, and combinations of two or more thereof. In an embodiment, the natural oil is selected from the group consisting of soybean oil, canola oil, linseed oil, and combinations thereof. In an embodiment, the natural oil is soybean oil. In an embodiment, the natural oil has a linolenic acid content of greater than 5 weight percent ("wt%").

[0009] In an embodiment, esterification of the natural oil is performed via transesterification. Transesterification can be performed using any conventional or hereafter discovered techniques. In an embodiment, the natural oil is transesterified via contact with an alcohol under transesterification conditions with either an acid or base catalyst. Glycerol byproduct is removed from the reaction products due to insolubility. The alcohol employed for transesterification is selected based on the desired alkyl substituent of the fatty acid alkyl esters. Alcohols suitable for use in transesterification include C_1 to C_8 monohydric linear alcohols, such as methanol, ethanol, propanol, and butanol, or C_3 to C_8 branched alcohols, such as isopropanol, isobutanol, and 2-

ethylhexanol. In an embodiment, the alcohol is methanol, such that the resultant fatty acid alkyl esters are fatty acid methyl esters.

[0010] In an embodiment, the fatty acid alkyl esters have the structure: R^1-C(=O)O-R^2, where R^1 is a linear or branched C_1 to C_8 alkyl group, and R^2 represents one or more of saturated, mono-unsaturated, and polyunsaturated C_{12} to C_{22} fatty acid chains.

[0011] A catalyst may also be employed for esterification (e.g., transesterification). Catalysts suitable for use in esterification include homogeneous alkali catalysts, including metal alkoxides such as sodium methoxide, potassium methoxide, and sodium ethoxide, or metal hydroxides such as potassium hydroxide, sodium hydroxide, or supported solid alkali catalysts. Other classes of catalysts that may also be employed include acids, acidic resins, double metal cyanide catalysts, enzymes, super acids, super bases, and metal salts. The catalyst can be in homogeneous or heterogeneous form. In an embodiment, the catalyst employed for transesterification is sodium methoxide solution in methanol.

[0012] Commercially available FAAEs may be employed in various embodiments. Examples of suitable commercially available FAAEs include SOYCLEAR™ 1500 and SOYGOLD™ 1100 (fatty acid methyl esters from soybean oil, available from AG Environmental Products, Inc.); CANOLAGOLD™ 110 (fatty acid methyl esters from canola oil, available from AG Environmental Products, Inc.); and SE-1885 and SE-1885D (fatty acid methyl esters from soybean oil, available from Felda Iffco, Inc.).

[0013] The fatty acid alkyl esters are then epoxidized via contact with an acid and an aqueous peroxide solution to thereby produce an epoxidized reaction mixture comprising epoxidized fatty acid alkyl esters, residual acid, residual peroxide, and water. Suitable peroxides for use in epoxidizing the natural oil include aqueous solutions of hydrogen peroxide, peroxycarboxylic acids, alkyl hydroperoxides, and tertiary hydroperoxides. In an embodiment, the peroxide employed is an aqueous solution of hydrogen peroxide.

[0014] Suitable acids for use in epoxidizing the fatty acid alkyl esters include carboxylic acids, such as formic acid and acetic acid; and peroxycarboxylic acids, such as performic acid and peracetic acid. In an embodiment, a peroxycarboxylic acid is employed, acting as both the acid and the peroxide. Catalysts such as mineral acids (e.g., sulfuric acid) and heterogeneous acid resins (e.g., Amberlite™ IR 120H, available from Rohm & Haas) may optionally be

employed in the presence of the acid. In an embodiment, the acid employed for epoxidation is formic acid.

[0015] In one or more embodiments, the epoxidation reaction is controlled so as to produce eFAAEs having an iodine value in the range of from 4 to 15 grams of iodine per 100 grams of epoxidized fatty acid alkyl esters ("g I_2 / 100 g"), in the range of from 4 to 10 g I_2 / 100 g, in the range of from 7 to 10 g I_2 / 100 g, or in the range of from 8 to 10 g I_2 / 100 g. Iodine value is determined according to the American Oil Chemists' Society ("AOCS") recommended practice Cd 1-25. Additionally, the controlled epoxidation reaction conditions can be selected so as to produce eFAAEs having an oxirane oxygen content of at least 6 wt%, or at least 6.5 wt%, based on the entire weight of the eFAAEs. In various embodiments, the eFAAEs can have an oxirane oxygen content up to 8 wt%, or 7.5 wt%, based on the entire weight of the eFAAEs. Oxirane oxygen content is determined according to AOCS recommended practice Cd 9-57.

[0016] Controlled epoxidation comprises selecting a combination of reaction temperature, reaction time, aqueous peroxide solution concentration, molar ratio of peroxide-to-carbon/carbon double bonds, and peroxide solution feed rate to achieve the desired iodine value and/or oxirane oxygen content. In an embodiment, the epoxidation reaction temperature employed can be maintained in the range of from 20 to 60 °C, in the range of from 30 to 50 °C, or in the range of from 40 to 50 °C. In various embodiments, the aqueous peroxide solution employed can have a concentration of less than 50 volume percent ("vol%"), less than 40 vol%, in the range of from 20 to 40 vol%, in the range of from 25 to 35 vol%, or of 30 vol%. In one or more embodiments, the molar ratio of peroxide-to-carbon/carbon double bounds in the FAAE can be from 1.5 to 2, from 1.7 to 2, or 2. In an embodiment, the peroxide solution feed rate can range from 0.2 to 2 grams of peroxide solution per gram of fatty acid alkyl esters per hour. In another embodiment, the peroxide solution feed rate can range from 0.3 to 4 moles of peroxide solution per molar equivalent of carbon-carbon double bonds in the fatty acid alkyl esters per hour. Regardless of which measurement is employed for determining the peroxide solution feed rate, in an embodiment, the peroxide solution feed rate can be controlled so that the epoxidation reaction temperature does not exceed the desired maximum temperature described above. In an embodiment, the peroxide feed rate can be controlled so as to prevent the epoxidation reaction temperature from exceeding 60 °C, 50 °C, or 40 °C.

[0017] In some embodiments, the reaction conditions chosen to maintain the above-described iodine value may cause decreased oxirane oxygen content. In order to achieve the desired oxirane oxygen content (e.g., at least 6 or at least 6.5 wt%), a longer-than-conventional reaction time may be employed. In various embodiments, the reaction time employed for controlled epoxidation can be greater than 6 hours, in the range of from 7 to 20 hours, in the range of from 8 to 15 hours, or in the range of from 10 to 12 hours.

[0018] Though not wishing to be bound by theory, it was surprisingly found that maintaining an iodine value of at least 4 produces eFAAE having low concentrations of hydrophilic impurities compared to conventional eFAAE. The term "hydrophilic impurities" denotes epoxidized fatty acid ester compounds containing hydroxyl groups formed from degraded epoxy rings on the fatty acid chain. In an embodiment, the eFAAE can have a hydrophilic impurities content of less than 0.8 wt%, less than 0.7 wt%, less than 0.6 wt%, less than 0.5 wt%, less than 0.4 wt%, less than 0.3 wt%, less than 0.2 wt%, or less than 0.1 wt% based on the entire weight of the eFAAE. Hydrophilic impurities content is determined by high performance liquid chromatography ("HPLC") according to the test method described in the following Examples.

[0019] Following epoxidation, the residual acid, peroxide, and water is removed from the epoxidized reaction mixture via layer separation and neutralization. Layer separation involves separation of an aqueous layer, which contains water, acids, peroxide, and possible traces of oil and esters, from an organic layer containing the eFAAE. To accomplish layer separation, the reaction mixture is allowed to settle and separate into two layers by density difference, and the bottom aqueous layer is disposed of while the top organic layer is processed further to obtain the desired product.

[0020] Following layer separation, the residual acid can be neutralized, such as by contact with a sodium/bicarbonate solution. Thereafter, the organic layer can be washed one or more times with water. In an embodiment, the organic layer is washed repeatedly until it is neutral (having a pH of about 7). Thereafter, the washed mixture can be subjected to layer separation again, followed by vacuum distillation of the top organic layer to remove residual water.

Plasticizer

[0021] The present disclosure provides a plasticizer composition comprising eFAAE, prepared as described above. Optionally, the plasticizer composition can further include other types of plasticizers, such as an eNO. Suitable epoxidized natural oils include epoxidized animal

and vegetable oils, such as epoxidized soybean oil ("eSO"), epoxidized corn oil, epoxidized sunflower oil, epoxidized palm oil, epoxidized linseed oil, epoxidized canola oil, epoxidized rapeseed oil, epoxidized safflower oil, epoxidized tall oil, epoxidized tung oil, epoxidized fish oil, epoxidized beef tallow oil, epoxidized castor oil, or combinations thereof. In an embodiment, the present plasticizer is a phthalate-free plasticizer, or is otherwise void or substantially void of phthalate.

[0022] When both eFAAE and eNO are present, the plasticizer composition can contain relative amounts of eFAAE (e.g., eFAME) to eNO (e.g., eSO) in a weight ratio in the range of from greater than (">") 0 : less than ("<") 100 to <100 : >0, more typically from 10:90 to 90:10, more typically from 20:80 to 80:20, and even more typically from 30:70 to 70:30. In another embodiment, the plasticizer composition comprises from 20 to less than 100 wt% eFAAE and from greater than 0 to 80 wt% eSO. Weight ratios and weight percents are based on total weight of the plasticizer composition. In various embodiments, the plasticizer composition consists of or consists essentially of eFAAE and eNO.

Polymeric Composition

[0023] The present disclosure provides a polymeric composition. In an embodiment, a polymeric composition is provided which includes a polymeric resin and the present plasticizer as disclosed above.

[0024] Non-limiting examples of suitable polymeric resins include polysulfides, polyurethanes, acrylics, epichlorohydrins, nitrile rubber, chlorosulfonated polyethylene, chlorinated polyethylene, polychloroprene, styrene butadiene rubber, natural rubber, synthetic rubber, EPDM rubber, propylene-based polymers, ethylene-based polymers, and vinyl chloride resins. The term, "propylene-based polymer," as used herein, is a polymer that comprises a majority weight percent polymerized propylene monomer (based on the total amount of polymerizable monomers), and optionally may comprise at least one polymerized comonomer. The term, "ethylene-based polymer," as used herein, is a polymer that comprises a majority weight percent polymerized ethylene monomer (based on the total weight of polymerizable monomers), and optionally may comprise at least one polymerized comonomer.

[0025] The term "vinyl chloride resin," as used herein, is a vinyl chloride polymer, such as polyvinyl chloride ("PVC"), or a vinyl chloride copolymer such as vinyl chloride/vinyl acetate copolymer, vinyl chloride/vinylidene chloride copolymer, vinyl chloride/ethylene copolymer or a

copolymer prepared by grafting vinyl chloride onto ethylene/vinyl acetate copolymer. The vinyl chloride resin can also include a polymer blend of the above-mentioned vinyl chloride polymer or vinyl chloride copolymer with other miscible or compatible polymers including, but not limited to, chlorinated polyethylene, thermoplastic polyurethane, olefin polymers such as a methacryl polymer or acrylonitrile-butadiene-styrene polymer.

[0026] In an embodiment, the polymeric resin is PVC.

[0027] In an embodiment, the polymeric composition includes from 25 wt% to 90 wt% PVC, from 5 wt% to 35 wt% eFAAE, from 0 wt% to 35 wt% eNO, and from 0 wt% to 35 wt% filler.

Additives

[0028] The polymeric composition may include one or more of the following optional additives: a filler, an antimicrobial agent, a biocide, a flame retardant, a heat stabilizer, an anti-drip agent, a colorant, a lubricant, a low molecular weight polyethylene, a hindered amine light stabilizer, a UV light absorber, a curing agent, a booster, a retardant, a processing aid, a coupling agent, an antistatic agent, a nucleating agent, a slip agent, a viscosity control agent, a tackifier, an anti-blocking agent, a surfactant, an extender oil, an acid scavenger, a metal deactivator, and any combination thereof.

[0029] In an embodiment, the polymeric composition includes PVC, the present plasticizer, a filler (calcium carbonate, clays, silica, and any combination thereof), metal soap stabilizers (zinc stearate or mixed metal soap stabilizers containing Ca, Zn, Mg, Sn, and any combination thereof), a phenolic or related antioxidant, and a processing aid.

Articles of Manufacture

[0030] Articles of manufacture can be prepared that comprise the above-described polymeric compositions. Such articles of manufacture can include those designed for use in the medical or food industries, particularly those articles that may frequently come into contact with water and where water-leachable compounds are a concern. Exemplary articles of manufacture include blood bags, intravenous bags, saline solution bags, syringes, intravenous tubing, nasogastric tubing, catheter tubing, drainage tubing, examination gloves, oxygen masks, orthodontic retainers, artificial skin, and food packaging (e.g., packaging for various beverages, meats, and frozen vegetables).

TEST METHODS

Hydrophilic Impurities Measurement

[0031] Quantify the amount of water-leachable (i.e., hydrophilic) impurities using HPLC with an evaporative light-scattering detector ("ELSD"). The method is as follows:

1) Add 0.04 g of eFAME (liquid) to 8 g of deionized water;
2) Heat the sample in an oven at 40 °C for 24 hours;
3) Remove 1 mL of the sample from the bottom of the vial;
4) Perform HPLC-ELSD for each sample.

HPLC setup:

[0032] Mobile phase: H_2O/Acetonitrile (A/B)

Column: ODS C-18; 2.1 x 100 mm, 3μm particle size

Gradient:

Time (min)	%B (Acetonitrile)
0.0	88
1.8	88
2.6	100
7.0	100
9.0	88
11.0	88

Flow: 0.30 ml/min.
Oven Temp.: 70 °C
Injection: 2 μl
Run Time: 11 min.
Post Time: 2 min.

ELSD setup:

[0033]
Instrument: Alltech 3300 ELSD
Tube Temp.: 70 °C
Gas Flow: 1.80 SLPM
Gain: 1.0
N_2 regulator: 60 psig

Oxirane Oxygen Content

[0034] Determine oxirane oxygen content according to AOCS Cd 9-57.

Iodine Value

[0035] Determine iodine value according to AOCS Cd 1-25.

EXAMPLES

Example 1

[0036] Charge 50 g of FAME (SOYCLEAR™ 1500, available from AG Environmental Products, Inc.) and 5.8 g of formic acid (98-100 % purity, obtained from RANKEM, RFCL Ltd.) to a 250-mL glass reactor equipped with an overhead stirrer having TEFLON™ blades and immersed in an oil bath having an initial temperature of 30 °C. The amounts of FAME and formic acid employed achieve an acid-to-carbon/carbon double bond ("C=C") mole ratio of 0.5. Add 57.5 g of 30 vol% hydrogen peroxide ("H_2O_2") (obtained from Merck & Co.) solution (in water), resulting in an H_2O_2-to-C=C mole ratio of 2. Add the H_2O_2 at a continuous rate for the initial 2 hours; maintain reaction temperature at 40 °C by adjusting the temperature of the oil bath. Agitate the reaction mixture via the overhead stirrer at 400 rpm to ensure proper mixing in the reactor. Maintain the reaction conditions for a total of 11 hours, including feeding time. After 11 hours, stop the agitation and allow the reaction mixture to separate into aqueous (bottom) and organic (top) layers over 2 hours. Drain the resulting aqueous layer to separate most of the water and formic acid. Neutralize the organic layer with dilute sodium bicarbonate solution (0.1 M solution prepared by dissolving 8.4 g of sodium bicarbonate powder obtained from S.d.fine Chem in 1 liter of distilled water), which essentially removes residual formic acid. A total of 75 mL of 0.1 M sodium bicarbonate solution is added in 5 steps for neutralization. Thereafter, wash the organic layer with water, repeating until it becomes neutral (approximately 25 mL of water, total). Measure the pH of the wash water after each wash using pH paper; continue washing until it reaches a pH value of about 7. After the last wash, add 50 mL of distilled water to a separating funnel containing the organic layer. Shake the mixture to ensure adequate contact and allow the mixture to settle. Once separation is achieved, drain the bottom aqueous layer. Place the top organic layer under vacuum (~10 mbar (1,000 Pa), 60 °C) for two hours to remove residual water.

Example 2

[0037] Prepare an eFAME sample as described above in Example 1, except employ FAME prepared via transesterification of soybean oil as the starting material. Prepare FAME according to the following method. Charge 100 g (0.113 moles) of soybean oil (obtained from Cargill Gemini) to a 500-mL, 3-neck, round-bottom flask. Add 21.72 g (0.68 moles) of methanol (99.5 % pure, obtained from Sigma Aldrich) to the reactor to maintain the mole ratio of

methanol:soybean oil at 6:1. The reactor is equipped with a condenser, temperature sensor, and overhead stirrer having TEFLON™ blades. The reactor is immersed in an oil bath to maintain the reaction temperature at 60 °C under nitrogen flow. Once the reaction temperature of 60 °C is achieved, 4 g of 25 % sodium methoxide dissolved in methanol (commercially available from Sigma Aldrich) is added to the reactor. After the reaction, wash the reaction mixture with water several times (till pH becomes about 7) to remove the residual sodium methoxide. Dry the final product under vacuum (10 mbar at 60 °C) to obtain FAME.

Example 3

[0038] Prepare an eFAME sample according to the method described in Example 1 using the starting material as prepared in Example 2, except maintain the epoxidation reaction temperature at 50 °C instead of 40 °C.

Comparative Example 1

[0039] Prepare and eFAME sample by charging 50 g of FAME (as prepared in Example 2) and 5.8 g of formic acid to a glass reactor (acid-to-C=C mole ratio of 0.5) equipped as described in Example 1. Add 34.5 g of 50 vol% H_2O_2 (obtained from Merck) solution (in water), giving an H_2O_2-to-C=C mole ratio of 2, and at a continuous rate while agitating the reaction mixture and maintaining the reaction temperature between 60 and 70 °C. After 6 hours of reaction time, stop the agitation and allow the reaction mixture to separate into aqueous and organic layers for 2 hours. The remaining steps are repeated as described in Example 1.

Comparative Example 2

[0040] Prepare and eFAME sample by charging 50 g of FAME (SOYCLEAR™ 1500) and 5.8 g of formic acid to a glass reactor (acid-to-C=C mole ratio of 0.5) equipped as described in Example 1. Add 34.5 g of 50 vol% H_2O_2 (obtained from Merck) solution (in water), giving an H_2O_2-to-C=C mole ratio of 2, and at a continuous rate while agitating the reaction mixture and maintaining the reaction temperature at 60 °C. After 3 hours of reaction time, stop the agitation and allow the reaction mixture to separate into aqueous and organic layers for 2 hours. The remaining steps are repeated as described in Example 1.

Analyses

[0041] For each of the samples prepared as described above, determine the oxirane oxygen content according to the above-described procedure. Additionally, determine the iodine number for each sample according to the above-described procedure. Finally, determine the hydrophilic

impurities content for each sample according to the above-described procedure. Results of these analyses are provided in Table 1, below.

Table 1 – Sample Properties

Property	Ex. 1	Ex. 2	Ex. 3	Comp. Ex. 1	Comp. Ex. 2
Oxirane oxygen (wt%)	6.51	6.50	6.97	6.09	6.74
Iodine number (g I_2 /100g)	8.29	9.91	4.16	1.65	3.19
Hydrophilic impurities (wt%)	0.081	0.083	0.14	1.6	0.98
Percent change in hydrophilic impurities relative to Comp. Ex. 2	-92	-92	-86	+64	-----

[0042] As can be seen from the results in Table 1, the amount of hydrophilic impurities in eFAME Examples 1 and 2 was reduced by more than 90 % when the iodine number was increased to between 8 and 10 from 3.2. The hydrophilic impurities content was also reduced by more than 85 % when the iodine number was increased to above 4 from 3.2, as seen in Example 3. On the other hand, when the iodine number was decreased further to 1.6, hydrophilic impurities increased by more than 60 %, as shown in Comparative Example 1.

CLAIMS

1. A plasticizer composition comprising:

epoxidized fatty acid alkyl esters,

wherein said epoxidized fatty acid alkyl esters have an iodine value in the range of from 7 to 10 g I_2 / 100 g of epoxidized fatty acid alkyl esters,

wherein said epoxidized fatty acid alkyl esters have an oxirane oxygen content of at least 6 weight percent based on the entire weight of said epoxidized fatty acid alkyl esters, and

wherein said epoxidized fatty acid alkyl esters are prepared from esterified soybean oil.

2. The plasticizer composition of claim 1, wherein said epoxidized fatty acid alkyl esters have a hydrophilic impurities content of less than 0.8 weight percent based on the entire weight of said epoxidized fatty acid alkyl esters.

3. A polymeric composition comprising a polymeric resin and the plasticizer composition of claim 1 or 2.

4. The polymeric composition of claim 3, wherein said polymeric resin is a vinyl chloride resin, wherein said polymeric composition is an article of manufacture selected from the group consisting of blood bags, intravenous bags, saline solution bags, syringes, intravenous tubing, nasogastric tubing, catheter tubing, drainage tubing, examination gloves, oxygen masks, orthodontic retainers, artificial skin, and food packaging.

5. A process for producing epoxidized fatty acid alkyl esters, said process comprising:

epoxidizing fatty acid alkyl esters via controlled epoxidation by contact with an acid and an aqueous peroxide solution to form epoxidized fatty acid alkyl esters,

wherein said controlled epoxidation comprises selecting a reaction temperature, a reaction time, an aqueous peroxide solution concentration, and a peroxide solution feed rate to cause said epoxidized fatty acid alkyl esters to retain sufficient unsaturation

to present an iodine value in the range of from 7 to10 g I_2 / 100 g of epoxidized fatty acid alkyl esters,

wherein said epoxidized fatty acid alkyl esters have an oxirane oxygen content of at least 6 weight percent based on the entire weight of said epoxidized fatty acid alkyl esters, and

wherein said fatty acid alkyl esters are prepared by transesterifying a soybean oil.

6. The process according to claim 5, wherein said epoxidized fatty acid alkyl esters have a hydrophilic impurities content of less than 0.8 weight percent based on the entire weight of said epoxidized fatty acid alkyl esters.

7. The process of claim 5 or 6, wherein said reaction temperature is in the range of from 30 to 50 °C, wherein said aqueous peroxide solution has a concentration of less than 40%, wherein said reaction time is greater than 6 hours, wherein said peroxide solution feed rate is in the range of from 0.3 to 4 moles of peroxide solution per molar equivalent of carbon-carbon double bonds in the fatty acid alkyl esters per hour.

8. The process of claim 5, 6 or 7, wherein said epoxidized fatty acid alkyl esters have the structure: R^1-C(=O)O-R^2, wherein R^1 is a linear or branched C_1 to C_8 alkyl group, and R^2 represents saturated, mono-unsaturated, and/or polyunsaturated C_{12} to C_{22} epoxidized fatty acid chains.