[0006] EP 601721, Ethyl Petroleum, June 15, 1994, discloses a process for preparing overbased phenates.

[0007] PCT publication WO 2013/119623, Lubrizol, August 15, 2013, discloses a sulfurized alkaline earth metal (e.g., calcium) dodecylphenate prepared by reacting dodecylphenol with calcium hydroxide or calcium oxide in an amount of about 0.3 to about 0.7 moles per mole of dodecylphenol charged and an alkylene glycol in an amount of about 0.13 to about 0.6 moles per mole of dodecylphenol charged; and reacting the product of the first step with sulfur in an amount of about 1.6 to about 3 moles per mole of dodecylphenol charged The product thus prepared has reduced levels of monomeric dodecylphenol.

[0008] The disclosed technology provides a method for preparing phenate detergent with improved filterability efficiency. In certain embodiments, the disclosed technology may also provide a product which contains a reduced amount of monomeric dodecylphenol within an oligomeric dodecylphenol composition.

SUMMARY OF THE INVENTION

[0009] The disclosed technology provides a process for preparing a sulfurized alkaline earth metal alkylphenate, optionally overbased, comprising:

(a) reacting (i) an alkylphenol, wherein the alkyl group contains 6 to 24 carbon atoms, with (ii) an alkaline earth metal hydroxide or an alkaline earth metal oxide in an amount of 0.4 to 10 moles per mole of alkylphenol charged to the reaction; and (iii) a sulfur source in an amount to provide 0.8 to 3 moles sulfur (as S) per mole of alkylphenol charged to the reaction; in the presence of (iv) an alkylene glycol or dialkylene glycol, or ether thereof, in an amount of 0.2 to 2 moles per mole of alkylphenol charged to the reaction; and including in the reaction mixture (v) an oil of lubricating viscosity; and optionally further reacting the product thereof with (vi) carbon dioxide; thereby forming a sulfurized alkaline earth metal alkylphenate composition in oil;

(b) heating said alkylphenate composition to 120 to 280°C or 200 to 250°C;

(c) supplying steam to said alkylphenate composition;

(d) removing said steam at a pressure lower than the steam is supplied in (c); and

(e) filtering the resulting composition to provide, as the filtrate, a sulfurized alkaline earth metal alkylphenate in oil.

[0009a] The invention also provides a method for improving the filterability of an alkaline earth metal in a process comprising: (a) reacting

(i) an alkylphenol, wherein the alkyl group contains 6 to 24 carbon atoms, with:

(ii) an alkaline earth metal hydroxide or an alkaline earth metal oxide in an amount of 0.4 to 10 moles per mole of alkylphenol charged to the reaction; and

(iii) a sulfur source in an amount to provide 0.8 to 3 moles sulfur (as S) per mole of alkylphenol charged to the reaction; in the presence of

(iv) an alkylene glycol or dialkylene glycol, or ether thereof, in an amount of 0.2 to 2 moles per mole of alkylphenol charged to the reaction; and including in the reaction mixture

(v) an oil of lubricating viscosity;

and optionally further reacting the product thereof with:

(vi) carbon dioxide;

thereby forming a sulfurized alkaline earth metal alkylphenate composition in oil;

(b) heating said alkylphenate composition to 120 to 280°C or 200 to 250°C;

said improvement comprising:

(c) supplying steam to said alkylphenate composition; and

(d) removing said steam at a pressure lower than the steam is supplied in (c); prior to

(e) filtering the resulting composition to provide, as the filtrate, a sulfurized alkaline earth metal alkylphenate in oil.

[0009b] A method for reducing the amount of monomeric alkylphenol in the product of a process comprising:

(a) reacting

(i) an alkylphenol, wherein the alkyl group contains 6 to 24 carbon atoms, with:

(ii) an alkaline earth metal hydroxide or an alkaline earth metal oxide in an amount of 0.4 to 10 moles per mole of alkylphenol charged to the reaction; and

(iii) a sulfur source in an amount to provide 0.8 to 3 moles sulfur (as S) per mole of alkylphenol charged to the reaction; in the presence of

(iv) an alkylene glycol or dialkylene glycol, or ether thereof, in an amount of 0.2 to 2 moles per mole of alkylphenol charged to the reaction; and including in the reaction mixture

(v) an oil of lubricating viscosity;

and optionally further reacting the product thereof with:

(vi) carbon dioxide;

thereby forming a sulfurized alkaline earth metal alkylphenate composition in oil;

(b) heating said alkylphenate composition to 120 to 280°C or 200 to 250°C;

said reduction arising from:

(c) supplying steam to said alkylphenate composition; and

(d) removing said steam at a pressure lower than the steam is supplied in (c); prior to

(e) filtering the resulting composition to provide, as the filtrate, a sulfurized alkaline earth metal alkylphenate in oil.

[0010] The disclosed technology further provides the product prepared by the foregoing process; a lubricant composition comprising an oil of lubricating viscosity and the

The Claims defining the invention are as follows:

1. A process for preparing a sulfurized alkaline earth metal alkylphenate, comprising:

(a) reacting

(i) an alkylphenol, wherein the alkyl group contains 6 to 24 carbon atoms, with:

(ii) an alkaline earth metal hydroxide or an alkaline earth metal oxide in an amount of 0.4 to 10 moles per mole of alkylphenol charged to the reaction; and

(iii) a sulfur source in an amount to provide 0.8 to 3 moles sulfur (as S) per mole of alkylphenol charged to the reaction; in the presence of

(iv) an alkylene glycol or dialkylene glycol, or ether thereof, in an amount of 0.2 to 2 moles per mole of alkylphenol charged to the reaction; and including in the reaction mixture

(v) an oil of lubricating viscosity;

and optionally further reacting the product thereof with:

(vi) carbon dioxide;

thereby forming a sulfurized alkaline earth metal alkylphenate composition in oil;

(b) heating said alkylphenate composition to 120 to 280°C or 200 to 250°C;

(c) supplying steam to said alkylphenate composition;

(d) removing said steam at a pressure lower than the steam is supplied in (c); and

(e) filtering the resulting composition to provide, as the filtrate, a sulfurized alkaline earth metal alkylphenate in oil.

2. The process of claim 1, wherein the alkylphenol comprises p-dodecylphenol.

3. The process of claim 1 or claim 2, wherein the amount alkaline earth metal hydroxide or oxide is 1 to 5 moles per mole of alkylphenol charged to the reaction.

4. The process of any one of claims 1 to 3, wherein the amount of alkaline earth metal hydroxide or oxide is suitable to provide a product with a total base number of 200 to 600 or 100 to 350 on an oil free basis, as measured according to ASTM D-2896.

5. The process of any one of claims 1 to 4, wherein the alkylene glycol or dialkylene glycol, or ether thereof, comprises ethylene glycol or propylene glycol.

6. The process of any one of claims 1 to 5, wherein the oil of lubricating viscosity is present at the time of removal of the steam in step (d) in an amount of 20 to 60 parts by weight per 100 parts by weight of alkylphenol charged to the reaction.

7. The process of any one of claims 1 to 6, wherein the steam is supplied at superatmospheric pressure and at a temperature of 120°C to 250°C.

8. The process of any one of claims 1 to 7, wherein volatile materials are removed along with removal of the steam.

9. The process of any one of claims 1 to 8, wherein the amount of steam is 3 to 36 parts by weight per 100 parts by weight of alkylphenol charged to the reaction.

10. A product prepared by the process of any one of claims 1 to 9.

11. A lubricant composition comprising an oil of lubricating viscosity and the product of claim 10.

12. A method for lubricating an internal combustion engine, comprising supplying thereto the lubricant composition of claim 11.

13. A method for improving the filterability of an alkaline earth metal in a process comprising:

(a) reacting

(i) an alkylphenol, wherein the alkyl group contains 6 to 24 carbon atoms, with:

(ii) an alkaline earth metal hydroxide or an alkaline earth metal oxide in an amount of 0.4 to 10 moles per mole of alkylphenol charged to the reaction; and

(iii) a sulfur source in an amount to provide 0.8 to 3 moles sulfur (as S) per mole of alkylphenol charged to the reaction; in the presence of

(iv) an alkylene glycol or dialkylene glycol, or ether thereof, in an amount of 0.2 to 2 moles per mole of alkylphenol charged to the reaction; and including in the reaction mixture

(v) an oil of lubricating viscosity;

and optionally further reacting the product thereof with:

(vi) carbon dioxide;

thereby forming a sulfurized alkaline earth metal alkylphenate composition in oil;

(b) heating said alkylphenate composition to 120 to 280°C or 200 to 250°C;

said improvement comprising:

(c) supplying steam to said alkylphenate composition; and

(d) removing said steam at a pressure lower than the steam is supplied in (c); prior to

(e) filtering the resulting composition to provide, as the filtrate, a sulfurized alkaline earth metal alkylphenate in oil.

14. The method of claim 13, wherein the filtering is accomplished by use of a filter aid and the amount of said filter aid used is less than 3 weight percent based on the final batch yield.

15. A method for reducing the amount of monomeric alkylphenol in the product of a process comprising:

(a) reacting

(i) an alkylphenol, wherein the alkyl group contains 6 to 24 carbon atoms, with:

(ii) an alkaline earth metal hydroxide or an alkaline earth metal oxide in an amount of 0.4 to 10 moles per mole of alkylphenol charged to the reaction; and

(iii) a sulfur source in an amount to provide 0.8 to 3 moles sulfur (as S) per mole of alkylphenol charged to the reaction; in the presence of

(iv) an alkylene glycol or dialkylene glycol, or ether thereof, in an amount of 0.2 to 2 moles per mole of alkylphenol charged to the reaction; and including in the reaction mixture

(v) an oil of lubricating viscosity;

and optionally further reacting the product thereof with:

(vi) carbon dioxide;

thereby forming a sulfurized alkaline earth metal alkylphenate composition in oil;

(b) heating said alkylphenate composition to 120 to 280°C or 200 to 250°C;

said reduction arising from:

(c) supplying steam to said alkylphenate composition; and

(d) removing said steam at a pressure lower than the steam is supplied in (c); prior to

(e) filtering the resulting composition to provide, as the filtrate, a sulfurized alkaline earth metal alkylphenate in oil.