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

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
15 December 2016 (15.12.2016)

WIPO | PCT

(10) International Publication Number
WO 2016/200941 A1

(51) International Patent Classification:
*C09G 1/02* (2006.01) *C09K 3/14* (2006.01)

(21) International Application Number:
PCT/US2016/036449

(22) International Filing Date:
8 June 2016 (08.06.2016)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
1020150132301 8 June 2015 (08.06.2015) BR

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

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

**Published**:

— *with international search report (Art. 21(3))*

WO 2016/200941 A1

(54) Title: LAPPING COMPOSITION FOR SUBSTRATES

(57) Abstract: The present invention refers to a polishing composition designed for conditioning surfaces, such as gears, seals, rings, and others. The composition is an oil in water emulsion, wherein the improvement was achieved when using a synergistic combination of additives, such as wetting agents, dispersants, surface tension modifiers and surface modifiers.

# LAPPING COMPOSITION FOR SUBSTRATES

The present invention refers to a lapping composition designed for conditioning surfaces such as gears, seals, rings, and others. The composition is an emulsion of water in oil wherein the improvement has been achieved when using a synergistic combination of additives, such as wetting agents, dispersants, surface tension modifiers and surface modifiers.

STATE OF THE ART

The lapidary composition industry has taken increasing proportions recently. This is due in part to the immense possibilities in the uses of lapidary compositions due to the great flexibility of their characteristics and features.

Lapping is an operation that uses low speed and low pressure in the finishing of metal parts, so as to correct minor imperfections on metal surfaces, typically by rubbing an abrasive composition against the substrate material in order to help condition the substrate surface.

Abrasive lapping processes in which the particulate abrasive is placed on the surface of a fabric are widely practiced. The particulate abrasive material is placed in a fluid carrier which suspends and transports the abrasive and lubricates the interface between the metallic shield and the workpiece to be conditioned so as to minimize metal to metal contact.

A mixture comprised of the carrier and the particulate abrasive is continuously fed to the surface of the metal shield and a uniform dispersion of the solid particles in the liquid carrier is desirable to achieve a controlled and uniform feeding to the interface between the metallic shield and the component being lapidated. The container containing the carrier and abrasive particle mixture is usually mixed with a stirrer so as to prevent solid particles from precipitating to the bottom of the reservoir.

Although it is known that the use of viscous media as the continuous phase tends to delay precipitation, highly viscous fluids are inadequate for the practice of most lapping operations in industry, in addition to being difficult to transport and to produce homogeneous mixtures.

In view of the above issues, it would be highly desirable to be able to achieve an improvement in the vehicles and particles, with the ability to maintain the abrasive particles in suspension at the working viscosity for extended periods of time without

external agitation. This fact would, without a doubt, be a major advance in the state of the art.

Documents which may be considered important in describing the state of the art regarding the technology developed herein for the present invention are patent documents US 2007/0251154 A1 and US 4,046,524.

Document US 2007/0251154 A1 relates to compositions of polishing compositions comprising a working fluid in a process for polishing the surface of an object, such process including contacting the surface of the object with one or more abrasives, in addition to contact with the polishing composition.

Document US 4.046.524 presents a lapping composition for use in polishing surfaces, such as gears, seals, rings and others, comprised of a hydrocarbon liquid that is gelled by adding a gelling agent, which is an alkyl-orthophosphate, to which a sandy abrasive is added.

## BRIEF DESCRIPTION OF THE INVENTION

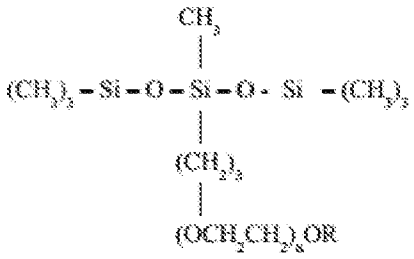
It is the objective of the present invention to provide a lapping composition capable of maintaining the abrasive particles suspended at the working viscosity for extended periods of time without external agitation. The present composition comprises a synergistic combination of additives, such as water, at least one organic solvent, at least one mineral oil and at least 1.8% by weight, based on the total mass of the composition, of a silicone polyether based stabilizer. Optional additives include wetting agents, dispersants, surface tension modifiers and surface modifiers.

## DETAILED DESCRIPTION OF THE INVENTION

Below, the object of the present invention will be described in detail, in an illustrative and not limiting manner, since the composition disclosed herein may contain different details and structural aspects without extrapolating the scope of protection claimed herein.

As mentioned above, the present invention relates to the composition of a lapping composition capable of keeping the abrasive particles in suspension at the working viscosity for extended periods of time without external agitation. The present composition comprises a synergistic combination of additives, such as at least one organic solvent, at least one mineral oil and at least 1.8% by weight, based on the total weight of the composition, of a silicone polyether based stabilizer. Optional additives include wetting agents, dispersants, surface tension modifiers and surface modifiers.

The present inventors have developed a composition surprisingly capable of maintaining stability for at least 10 days at room temperature. The composition of the lapping composition in the present invention comprises at least 1.8% by weight, based on the total weight of the composition of a silicone polyether based stabilizer. Preferably, the silicone polyether is selected from the family of trisiloxanes.



Commercially available examples of such silicone polyether include XIAMETER® OFX-5211 from Dow Corning, Hortolândia, Brazil.

The unexpected results obtained with this composition are partly produced by silicone polyether. The inventors of the present composition postulate that the silicone polyether produces a "super-spreading" of the generally hydrophobic abrasive particles present in the composition. Thus, it is believed that the silicone polyether present in the composition promotes super-spreading of the mineral oil over the surface of the particles in suspension (especially silicon carbide, which is non-polar), resulting in a greater packing of these particles and thus a larger packing volume fraction. As this situation leads to a weaker ordered structure, the composition flows with greater difficulty at steady state, resulting in high "yield stress" and greater stability to sedimentation.

The present composition optionally comprises at least one organic solvent to facilitate ring cleaning after using the present composition in the lapping process. Examples of solvents that may be used in the present composition include, but are not limited to, isoparaffin, dodecane, thinners, degreasers, etc.

The present composition further comprises a mineral oil that serves as lubricant and vehicle for mineral suspension. Examples of mineral oils that can be used in the present composition include, but are not limited to, USP 70 white mineral oil, paraffin oil, USP 90 mineral oil etc.

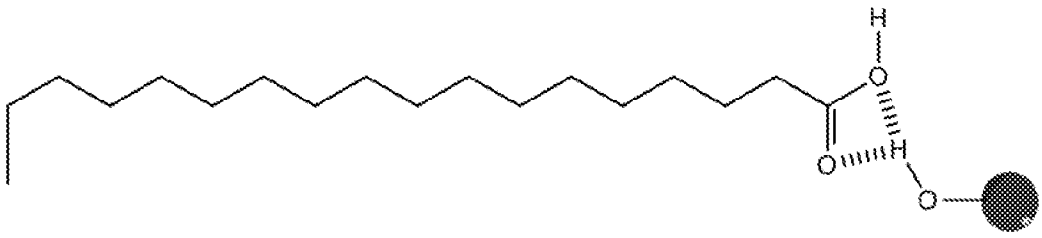
Lapping compositions generally comprise an abrasive mineral. Any abrasive mineral may be used in the composition of the composition subject of the patent application, such as

alumina, silicon carbide, chromium oxide, calcined bauxite, magnesium silicate, and iron oxide, among others. In one embodiment, the abrasive mineral is selected from the group consisting of aluminum oxide and silicon carbide. Commercially available examples of abrasive minerals include TAP-8 and EC6R 600F, both by Imerys (Salto, Brazil).

The composition of the present invention optionally comprises a colloidal silica based thickener to adjust the viscosity to a range suitable for working. Examples of thickeners available include products from the Aerosil family by Evonik (Americana, Brazil) and Cab-o-Sil by Cabot (Boston, USA).

Optionally, the present invention comprises a second aqueous stabilizer based on fatty alcohol ethoxylate and a fatty acid. Such stabilizers aid in the dispersion of other minerals optionally present in the composition, and that may not interact with the silicone polyether, such as alumina, for example. Commercially available examples of the second stabilizer include Tergitol 15-S-7 by Dow Chemicals (Jundiai, Brazil) and Quimipel Coat 9330 by Quimipel Indústria Química (Piracaia, Brazil).

It is believed that the second stabilizer, being a bidentate chelating agent, is useful in the stabilization of the functional groups on the surface of the alumina, as shown in the figure below.




Prior state of the art compositions aimed at increasing the stability of the composition by adding thickeners. However, this practice results in an increase in viscosity that makes it difficult to apply the composition using a pumping system.

The inventors of the present invention have developed a composition whose viscosity remains equal to or lower than 2500 cP at a temperature of 25°C. This specified viscosity range is important, not only for maintaining the minerals in suspension, but also to facilitate the application of the liquid through the pumping system.

The composition of the present invention has between 50% and 100% stabilization of solid particles in suspension, and such stabilization of between 50% and 100% is maintained without external agitation or shearing for up to 10 days.

The additives comprised in the composition as shown above, acted extremely synergistically with respect to the stabilization of the mineral mixture in suspension. As the silicone polyether acts by stabilizing the non-polar mineral oil (silicon carbide), the other stabilizers may act on the other components of the composition, for example, a polar mineral (alumina), resulting in a mixture synergistically stable, even at low viscosities.

The present invention has as its main advantages:

- The metal part polished with the present composition may be quite easily cleaned with either organic solvents or with water. This is possible because in addition to being a mineral suspension in mineral oil, the composition is also a water in oil emulsion. The water in question comes from the fatty acid used;

- The composition provides kinetic stability of the highly improved suspension for the abrasive particles; and

- The composition provides highly desirable flow characteristics, not only under shear, but also in a static system, resulting in unexpected improvements in the lapping rate.

EXAMPLES

TESTS:

Density: the density of each polishing composition prepared as described below was measured using a 25 mL polished stainless steel pycnometer with lid, following the procedure described in technical standard ASTM D1475 - 13, "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products".

Total Solids: total solids was determined for each lapping composition using a total solids analyzer supplied by MARTE (Sao Paulo, SP, Brazil), based on the mass loss after gradually heating the sample under infrared radiation, to a constant mass (heating ramp from 30 to 250°C in 15 min). The mass of the lapping composition samples was determined before the test and after heating the samples. The mass loss was then calculated as the total percentage of lost mass.

Stability: the stability of each sample was estimated using the dynamic rheology technique in the linear viscoelastic region, utilizing a DHR3 TA Instruments

brand rheometer. The parameter "yield stress" was calculated using the software available with the rheometer (Trios Software). Test conditions:

Conditioning sample:

- Temperature 25°C inherited set point: off
- Immersion time 10.0 s - wait for the temperature: Off
- Waiting for axial force: Off Pre-shear performance On shear rate 11/s
- Duration: 10.0 s
- Equilibrium performance: On Duration 120.0 s

Flow Scan:

- Temperature 25°C inherited set point: Off
- Immersion time 0.0 s waiting for temperature: Off
- Logarithmic Scan
- shear rate: 0.01 to 1001/sec
- Points per decade: 5
- Steady State Sensor: On Maximum equilibrium time 180.0 s
- Sample Period: 30.0 s
- % tolerance: 5.0
- Consecutive within 3
- Average graduated time: Off

Final test conditioning:

- Reference Temperature: On Temperature 25°C
- Idle system reference temperature (only if axial force control is active): Off
- Data Run: 3/18/2015
- Geometry Name: parallel plates of 25 mm, ETC Steel - 100577

- TA version of Trios Instruments: 3.1.4.3607

Materials:

| Name / Trade name | Description | Supplier |
|---|---|---|
| 70USP Grade | Mineral oil | Agecom, Mauá-SP |
| Isoparaffin 17/21 | Isoparaffin | Unipar, São Paulo-SP |
| TERGITOL 15-S-7 | Non-ionic, secondary fatty alcohol ethoxylate based surfactant | Dow Chemicals, Jundiaí-SP |
| XIAMETER® OFX-5211 | Non-ionic silicone polyether based surfactant | Dow Corning, Hortolandia-SP |
| AEROSOL OT-75% | Non-ionic sodium dialkyl-sulfosuccinate based emulsifier | Allnex Brs. Prod. Quim., Ponta Grossa-PR |
| QUIMIPEL COAT 9330 | Fatty acid emulsion | Quimipel Ind. Quim., Piracaia-SP |
| TAP 8 | Aluminum oxide | Imerys Fused Minerals, Salto-SP |
| EC6R 600F | Silicon carbide | Imerys Fused Minerals, Salto-SP |
| CAB-O-SIL M5 | Colloidal silica | Bandeirante Quimica, Mauá-SP |
| Tetronic 1107 | Non-ionic EO-PO block copolymer based surfactant | BASF, São Paulo-SP |
| PIB-32 | Polyisobutylene | Braskem, Camaçari-BA |

Experimental procedure:

COMPARATIVE EXAMPLES A-H AND EXAMPLES 1-15

The lapping composition of Comparative Examples A-H and Examples 1-7 were prepared using the following experimental procedure: the materials listed in Table 1 were added to a clean, dry flask in the order presented. The mixture was homogenized using a Cowles mechanical stirrer (Siemens Model ML-10, supplied by Tedemix, São Paulo, SP) at room temperature until the formation of a vortex in the center of the mixture and for at least 10 min. The amount of each ingredient is expressed in Table 1 as percent by weight (% w), based on the total percentage of the composition (100%).

Table 1

| Examples | % m | | | | |
|---|---|---|---|---|---|
| | Mineral oil | Isoparaffin | Tergitol 15-S-7 | XIAMETER® OFX-5211 | Aerosol OT-75 |
| Example 1 | 31.2 | 7.8 | 1.1 | 1.8 | 4.5 |
| Example 2 | 35 | 7.8 | 0.3 | 1.8 | 1.5 |
| Example 3 | 7.7 | 7.8 | 0.3 | 1.8 | 1.5 |
| Example 4 | 6.9 | 7.8 | 1.1 | 1.8 | 1.5 |
| Example 5 | 4.7 | 7.8 | 0.3 | 1.8 | 4.5 |
| Example 6 | 31.2 | 7.8 | 1.1 | 1.8 | 4.5 |
| Comparative Example A | 20.05 | 7.8 | 0.7 | 1.2 | 3 |
| Comparative Example B | 20.05 | 7.8 | 0.7 | 1.2 | 3 |
| Comparative Example C | 8.1 | 7.8 | 1.1 | 0.6 | 1.5 |
| Comparative Example D | 5.9 | 7.8 | 0.3 | 0.6 | 4.5 |
| Comparative Example E | 33.2 | 7.8 | 0.3 | 0.6 | 4.5 |
| Comparative Example F | 20.05 | 7.8 | 0.7 | 1.2 | 3 |
| Comparative Example G | 36.2 | 7.8 | 0.3 | 0.6 | 1.5 |
| Comparative Example H | 5.1 | 7.8 | 1.1 | 0.6 | 4.5 |
| Comparative Example I | 35.4 | 7.8 | 1.1 | 0.6 | 1.5 |

After homogenizing the composition, the desired amount of fatty acid emulsion was added slowly under mechanical agitation, as shown in Table 2. After completing the addition, the mixture was stirred again for at least 30 min at room temperature.

With the aid of a spatula, the desired amount of aluminum oxide (alumina) was slowly added to the mixture using mechanical agitation, followed by the silicon carbide, as shown in Table 2. After the addition was complete, the composition was kept under agitation for at least 30 min. at room temperature, to ensure adequate dispersion of the minerals.

Small quantities of colloidal silica were then slowly added under constant mechanical stirring, until each composition had a viscosity of about 2500 cP (approximately 0.10% in each formulation), as indicated in Table 2. The composition was stirred for about 30 minutes.

Table 2

| Examples | % m | | | |
|---|---|---|---|---|
| | **Fatty acid (QUIMIPEL COAT 9330)** | **Aluminum oxide (TAP08)** | **Silicon carbide (EC6R 600F)** | **Colloidal silica (CAB-O-SIL)** |
| Example 1 | 13.7 | 32.1 | 7.8 | 0.10 |
| Example 2 | 13.7 | 32.1 | 7.8 | 0.10 |
| Example 3 | 41 | 32.1 | 7.8 | 0.10 |
| Example 4 | 41 | 32.1 | 7.8 | 0.10 |
| Example 5 | 41 | 32.1 | 7.8 | 0.10 |
| Example 6 | 13.7 | 32.1 | 7.8 | 0.10 |
| Example Comp. A | 27.35 | 32.1 | 7.8 | 0.10 |
| Example Comp. B | 27.35 | 32.1 | 7.8 | 0.10 |
| Example Comp. C | 41 | 32.1 | 7.8 | 0.10 |
| Example Comp. D | 41 | 32.1 | 7.8 | 0.10 |
| Example Comp. E | 13.7 | 32.1 | 7.8 | 0.10 |
| Example Comp. F | 27.35 | 32.1 | 7.8 | 0.10 |
| Example Comp. G | 13.7 | 32.1 | 7.8 | 0.10 |
| Example Comp. H | 41 | 32.1 | 7.8 | 0.10 |
| Example Comp. I | 13.7 | 32.1 | 7.8 | 0.10 |

The viscosity of the composition was measured with a Brookfield viscometer (DV-II + Pro model) using number 03 spindle and a 30 rpm stirrer speed. The final viscosity of each composition is shown in Table 3.

The lapping composition in Examples 1-15 were subjected to density, total solids and stability measurements, following the experimental procedure described above. Results are reported in Table 3, below.

"Yield Stress" measurements, obtained using an RH-3 Discovery TA Instruments (New Castle, DE, USA) rheometer, and calculated according to the Waal model, represents the minimum pressure on the liquid so that it flows. Therefore, the lower the yield stress value, the greater the tendency of liquid to flow or, in other words, decant. Therefore, the lower the "yield stress" the more unstable the colloidal dispersion will be. The stability of each composition was determined based on the Yield Stress values and visual observation of each sample. Samples decanting in less than 10 days

were classified as "sedimented". The lapping compositions of the present invention had their stabilities classified as "stable" in the examples in which no decantation was observed within up to 10 days.

Table 3

| Examples | Density (g / ml) | Total Solids (%) | Yield Stress (Pa) | Stability |
|---|---|---|---|---|
| Example 1 | 1.3 | 91.48 | 6.27 | Stable |
| Example 2 | 1.4 | 88.9 | 6.06 | Stable |
| Example 3 | 1.5 | 67.23 | 14.97 | Stable |
| Example 4 | 1.5 | 66.38 | 6.88 | Stable |
| Example 5 | 1.4 | 67.67 | 20.72 | Stable |
| Example 6 | 1.4 | 83.79 | 4.52 | Stable |
| Ex. Comp. A | 1.6 | 76.93 | 1.4 | Sedimented |
| Ex. Comp. B | 1.5 | 80.53 | 1.28 | Sedimented |
| Ex. Comp. C | 1.3 | 69.38 | 0.97 | Sedimented |
| Ex. Comp. D | 1.5 | 69.83 | 0 | Sedimented |
| Ex. Comp. E | 1.4 | 81.72 | 0 | Sedimented |
| Ex. Comp. F | 1.5 | 83.55 | 1.18 | Sedimented |
| Ex. Comp. G | 1.5 | 83.59 | 0 | Sedimented |
| Ex. Comp. H | 1.7 | 69.68 | 3.86 | Sedimented |
| Ex. Comp. I | 1.5 | 84.53 | 3.45 | Sedimented |

COMPARATIVE EXAMPLES I-V

Comparative lapping compositions were prepared following the procedure described above for Comparative Examples A-H and Examples 1-7 but using a different stabilizing system. In the present description, a mixture of EO-PO block copolymer (Tetronic 1107 nonionic surfactant, BASF, São Paulo) and polyisobutylene (PIB-32, Braskem Camaçari) was used. The amount of each ingredient is expressed in Table 4 as percent by weight (% w), based on the total percentage of the composition (100%).

Table 4

| Examples | % m | | | | | | |
|---|---|---|---|---|---|---|---|
| | Mineral oil | Iso-paraffin | TETRONIC 1107 | PIB-32 | TAP08 | EC6R 600F | CAB-O-SIL |
| Comp. Example J | 28.7 | 28.7 | 1.25 | 1.25 | 32.1 | 7.7 | 0.3 |
| Comp. Example L | 29.45 | 29.45 | 0.5 | 0.5 | 32.1 | 7.7 | 0.3 |
| Comp. Example M | 28.7 | 28.7 | 1.25 | 1.25 | 32.1 | 7.7 | 0.3 |
| Comp. Example N | 27.95 | 27.95 | 2 | 2 | 32.1 | 7.7 | 0.3 |

| Examples | % m | | | | | | |
|---|---|---|---|---|---|---|---|
| | Mineral oil | Iso-paraffin | TETRONIC 1107 | PIB-32 | TAP08 | EC6R 600F | CAB-O-SIL |
| Comp. Example O | 28.7 | 28.7 | 0.5 | 2 | 32.1 | 7.7 | 0.3 |
| Comp. Example P | 28.7 | 28.7 | 1.25 | 1.25 | 32.1 | 7.7 | 0.3 |
| Comp. Example Q | 28.7 | 28.7 | 2 | 0.5 | 32.1 | 7.7 | 0.3 |
| Comp. Example R | 29.23 | 29.23 | 0.19 | 1.25 | 32.1 | 7.7 | 0.3 |
| Comp. Example S | 28.17 | 28.17 | 2.31 | 1.25 | 32.1 | 7.7 | 0.3 |
| Comp. Example T | 29.23 | 29.23 | 1.25 | 0.19 | 32.1 | 7.7 | 0.3 |
| Comp. Example U | 28.17 | 28.17 | 1.25 | 2.31 | 32.1 | 7.7 | 0.3 |
| Comp. Example V | 28.7 | 28.7 | 1.25 | 1.25 | 32.1 | 7.7 | 0.3 |
| Comp. Example X | 28.7 | 28.7 | 1.25 | 1.25 | 32.1 | 7.7 | 0.3 |

The stability of each composition was determined based on visual observation of each sample. Samples where decantation was observed in less than 12 hours had their stability classified as "very poor" or "poor" (decantation in less than 24 hours). The lapping compositions of the present invention had their stabilities classified as "good" (no decantation observed after 72 hours) or "excellent" (no decantation observed after 120 hours).

Table 5

| Examples | Stability |
|---|---|
| Comparative Example J | Poor |
| Comparative Example L | Poor |
| Comparative Example M | Poor |
| Comparative Example N | Poor |
| Comparative Example O | Poor |
| Comparative Example P | Poor |
| Comparative Example Q | Poor |
| Comparative Example R | Poor |
| Comparative Example S | Poor |

| Examples | Stability |
|---|---|
| Comparative Example T | Poor |
| Comparative Example U | Poor |
| Comparative Example V | Poor |
| Comparative Example X | Poor |

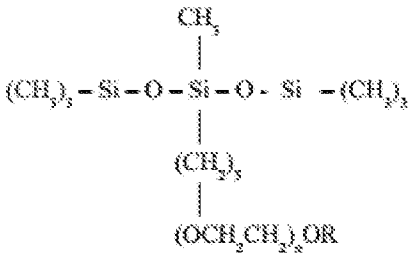
Thus, it can be concluded that the embodiments of the present invention solve the current state of the art problems, by providing an entirely new composition that results in a product with unique features not observed so far in similar products known.

These and other variations and modifications in the description will become apparent to those skilled in the art without diverting from the scope of the description, and it should be understood that this disclosure is not limited to the illustrative forms set forth herein.

## CLAIMS

1. A composition for lapping substrates containing at least one abrasive mineral in a mixture of water and at least one mineral oil, said composition being characterized by comprising at least 1.8% by weight, based on the total mass of the composition, of a silicone polyether based stabilizer.

2. A composition according to claim 1, characterized by silicone polyether being selected from the trisiloxane family.



3. A composition according to claim 1, characterized by further comprising a colloidal silica based thickener.

4. A composition according to claim 1, characterized by the abrasive mineral being selected from the group consisting of aluminum oxide and silicon carbide.

5. A composition according to claim 1, characterized by further comprising a second stabilizer based on fatty alcohol ethoxylate and a fatty acid.

6. A composition according to claim 1, characterized by said composition having a viscosity of between 1,000 and 20,000 cP at a temperature of 25°C.

7. A composition according to claim 2, characterized by presenting between 50% and 100% stabilization of solid particles in suspension.

8. A composition according to claim 6, characterized by the stabilization of between 50% and 100% being maintained without external agitation or shearing for at least 10 days.

9. A composition according to claim 1, characterized by additionally comprising at least one organic solvent.

**INTERNATIONAL SEARCH REPORT**

International application No.
**PCT/US2016/036449**

**A. CLASSIFICATION OF SUBJECT MATTER**

**C09G 1/02(2006.01)I, C09K 3/14(2006.01)I**

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)
C09G 1/02; C11D 3/36; C11D 1/72; C11D 3/37; C23C 26/00; C11D 3/26; C11D 1/08; C11D 17/00; C09K 3/14

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
eKOMPASS(KIPO internal) & Keywords: abrasive, polishing, lapping, lapidary composition, superwetting agent, silicon polyether, Dow Corning OFX-5211, Q2-5211

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | US 2007-0275867 A1 (SEROBIAN, A. K.) 29 November 2007<br>See paragraphs [0138], [0141], [0175]; Examples 43-47; claims 1, 2, 12; Tables 4-6. | 1-9 |
| A | US 6090765 A (BLACK, R. H. et al.) 18 July 2000<br>See Tables 2-4; claim 1. | 1-9 |
| A | WO 98-17765 A1 (AGENCY DESIGN SERVICES LIMITED) 30 April 1998<br>See Examples 7, 8, 14. | 1-9 |
| A | US 2009-0035451 A1 (SEROBIAN, A.) 05 February 2009<br>See paragraphs [0033], [0062], [0063], [0068]; Examples 1, 2; Tables 1, 6; claims 1, 9-11. | 1-9 |
| A | US 2008-0171683 A1 (JOHNSON, A. K. et al.) 17 July 2008<br>See paragraph [0066]; claim 1. | 1-9 |

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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| 24 August 2016 (24.08.2016) | **24 August 2016 (24.08.2016)** |


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**INTERNATIONAL SEARCH REPORT**

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**PCT/US2016/036449**

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