United States Patent [19]
Walton et al.

[11] 3,893,962
[45] July 8, 1975

[54] TELEPHONE CABLE FILLING COMPOSITION (II)

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[22] Filed: Jan. 7, 1974

[21] Appl. No.: 431,160

[52] U.S. Cl. ..... 260/28.5 A; 117/232
[51] Int. Cl. C08f 45/52
[58] Field of Search ..... 260/28.5 A

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[57] ABSTRACT

A composition suitable for filling telephone cables if described, which comprises:

1. a liquid polybutene of average molecular weight in the range of 500 –3500;

2. microcrystalline slackwax; or an equivalent thereof, comprising a mixture of a microcrystalline wax with brightstock oil or a high viscosity mineral oil of comparable physical properties;

3. polyethylene, of average molecular weight in the range of 10,000 – 20,000;

4. at least one wax different from (2) above, chosen from (a) paraffin waxes having melting points in the range of 37.8° – 79.5°C, (b) synthetic Fischer-Tropsch type waxes having melting points in the range of 71.1° – 115°C., and (c) natural waxes selected from the group: Beeswax, Carnauba wax, Chinese insect wax, Japan wax, Myrtle wax and Spermaceti wax; and (5) finely divided silica. The amount of finely divided silica used ranges from about 0.5% to about 8.0%, and preferably 0.5% – 5%, by weight of the composition. A larger proportion of polybutene is employed in the composition than that of any other ingredient. The polybutene, microcrystalline slackwax, polyethylene and

the second-mentioned wax are blended together and heated to about 125°C. and the batch stirred until the waxes and polyethylene are completely dissolved. Then the finely divided silica (silica flour) is added in small increments and thoroughly dispersed with high speed, high shear stirring until complete uniformity of the composition is attained.

32 Claims, No Drawings

TELEPHONE CABLE FILLING COMPOSITION (II)

This invention relates to compositions for the filling of telecommunications cables, particularly of buried cables which are subject to the ingress of water, or to entry and condensation of water vapor. Such water might flow through the cable and degrade its electrical properties.

The telecommunications cables to which the compositions of this invention are applicable are of the type comprising a multiplicity of conductors each having a dielectric of plastic material, a waterproof sheath enclosing the insulated conductors and, filling the interstices between these insulated conductors and between them and the cable sheath from end to end of the cable length, a water-impermeable medium which will not drain under the influence of gravity or such hydrostatic pressure as may arise in the event of damage to the cable sheath but which will permit relative sliding movement of the plastic insulated conductors over one another during such bending of the cable as occurs during manufacture and installation of the cable.

Water or water vapor often enters a cable through punctures in the cable's outer jacket. These punctures may be the result of lightning strikes, mechanical damage to the cable sheath, or of initial defects incurred during production or laying of cable. One way which has been employed to minimize water ingress is to sheath the cable interior with water and vapor barriers. Such barriers, however, are expensive. Also, such barriers, once they are penetrated, permit the entry of water which flows along the cable through interstices between the cable's conductors, fills the cable, and deteriorates its electrical qualities. Such deterioration manifests itself as an increase in the capacitance between cable conductors and results in increased losses. In telephone communication cables such losses can seriously degrade the operating performance of a telephone system. As will be appreciated, water (with a dielectric constant of 80) in the cable would increase signal losses to such an extent that transmission would virtually cease or alternatively the signals would become so garbled as to become meaningless. Ultimately, the water in the cable may corrode the conductors so as to cause open circuits.

It is known to fill the interstices of multiconductor telephone cables with water blocking compounds in order to prevent water introduced into the cables which are installed in ducts or directly buried in the ground, as a result of damage to the cable sheath, travelling along the interior of the cable from the point of entry and thus adversely affecting its electrical characteristics along its whole length. Various blocking compounds are known for this purpose, among them being heavy bodied mastic materials and jelly-like substances, such as petroleum jelly.

The material used in the substance filling the interstices is preferably of such a consistency that when applied to the cable, the substance adheres to the conductors and the sheath well enough to prevent the formation of water passages along the surfaces of the conductors or the inner surfaces of the sheath and also does not significantly reduce the flexibility of the cable. It is also important that the water-blocking compound be of such a consistency at the cable operating temperature that in the event of damage to the cable sheath it will not exude from the cable thus permitting the entry of water into the cable.

Various compositions for filling telecommunications cables to inhibit or prevent the ingress of water thereinto are known. Examples of such compositions are those comprising mixtures of a mineral oil and microcrystalline wax and/or synthetic hydrocarbon waxes, as described, for instance, in Albert King's U.S. Pat. Nos. 2,914,430 and 2,956,036, and in British Pat. Specifications Nos. 877,895 and 955,348 of Sargent et al. These and other known cable filling compositions do work reasonably well in fulfilling their intended purpose and have been widely used; but nevertheless have certain drawbacks.

More specifically, previously known telecommunications cable filling compositions have one or more of the following drawbacks:

a. Poor compatibility with the plastic materials used for insulating the cable pairs.

b. Frequently previous compounds were of low melting point and poor cohesive (internal) strength which render them unsuitable for use in cables where high temperatures could be met in service.

c. Excessive hardness at low temperature with the concomitant difficulty of handling filled telephone cables during cold weather installation.

d. Poorly chosen ingredients, some or all of which often exhibit exceptionally harmful effects on the plastic materials used to insulate the cable pairs (see (a) above).

e. The tendency for the components to separate (i.e. syneresis) due to poorly chosen base oils/polymers or improper blending techniques.

f. Low resistance to manual working resulting in a physical degradation of the composition to the point where its high temperature characteristics, especially drainage and water barrier characteristics, are adversely affected.

The ideal characteristics one should look for in a telephone cable filling composition for use in conditions where the cable may be subjected to a wide range of temperature during operation can be listed as follows:

1. It should have a high melting point, that is, above 80°C.; so that, when it is subjected to high ambient temperatures such as may be encountered during operation of the cable, it will not liquefy and thus tend to exude from the cable.

2. It should exhibit plasticity at very low temperatures, for example, at −40°C.

3. It should exhibit a minimum degree of syneresis, i.e. separation of liquid components from solid components.

4. It should exhibit minimum void formation on contraction.

5. It should exhibit a minimum change in dielectric constant with increase in frequency.

6. It should have a low value for the dielectric constant (permittivity) -between 2.0 and 2.4.

7. It should have maximum compatibility with primary insulating materials, such as plastics which are used for the cable sheathing and for enclosing the conductors.

8. It should have a liquid viscosity low enough that the cable interstices can be readily impregnated during the cable manufacturing operation, yet sufficiently high that the material would not exude from the cable dur-

ing manufacture or in the event of damage to the cable sheath.

9. It should be free from moisture and other polar contaminants, all of which have an adverse effect on the performance of the finished telephone cable.

10. It should have sufficient internal strength or "internal cohesive force" to withstand the pressure effect of a moderately high head of water at elevated temperatures, i.e. significantly above ambient temperatures, e.g. 50° – 80°C.

Known filling materials for use in cables, and in particular, those heretofore used in multiconductor telecommunications cables are deficient in one or more of the above characteristics. Thus there has been a continuing search for improved cable filling compositions which would overcome the deficiencies of known compositions of this type, and have characteristics more nearly approaching the ideal characteristics noted above.

An objective of the present invention is to provide cable filling compositions which substantially eliminate, or at least minimize, the ingress of water into telecommunications cables in the event of mechanical or electrical damage to the cable sheaths.

Another objective of the invention is to provide cable filling compositions which are an improvement over known cable filling compounds, in that they are free from at least some of the drawbacks of the known compounds, and which at least approach the ideal for compositions of this type.

We have found that these objectives can be fulfuilled by providing a composition which comprises, broadly, a mixture of (1) a low molecular weight, liquid polybutene; (2) microcrystalline slackwax, or an equivalent thereof comprising a mixture of microcrystalline wax with a high viscosity mineral oil such as brightstock oil; (3) polyethylene; (4) at least one wax of a type different from that in (2) above; and (5) finely divided silica; the silica being present in an amount ranging from about 0.5% to about 8.0% by weight of the composition. The finely divided silica serves as a filler and also as a gelling agent for binding the other ingredients into a gel structure.

Thus in one broad aspect the present invention resides in a composition suitable for use in filling telecommunications cables and te like, commprising: (1) polybutene, of average molecular weight in the range of 500–3500; (2) microcrystalline slackwax; or an equivalent thereof, comprising a mixture of at least one mcrocrystalline wax with a high viscosity mineral oil having the following characteristics:

Viscosity/98.8°C (ASTM D2161) — 120 SUS — 220 SUS

Viscosity/37.8°C (ASTM D2161) — 2000 SUS — 3500 SUS

Flash Point — 204.4°C. Minimum

Specific Gravity/15.6°C — 0.875 – 0.925

Colour ASTM 2–ASTM 8

Boiling Range (at atmospheric pressure) — 260° – 704.5°C.; (3) polyethylene, of average molecular weight in the range of 10,000 to 20,000; (4) at least one wax selected from the group consisting of (a) paraffin waxes having melting points in the range 37.8° – 79.5°C. (b) synthetic Fischer-Tropsch type waxes having melting points in the range of 71.7° – 115°C, and (c) natural waxes selected from the group: Beeswax, Carnauba wax, Chinese insect wax, Japan wax, Myrtle wax and Spermaceti wax; and (5) finely divided silica, said silica being present in an amount ranging from about 0.5% to about 8.0% by weight of said composition; said composition containing a larger proportion by weight of said polybutene than that of any other ingredient.

The telephone cable filling compositions of this invention are designed to fill the cable in such a manner that in the event of sheath failure water will not penetrate into the interior of the cable, thereby damaging or even destroying completely the transmission characteristics of the cable. The compositions of the present invention are designed to have sufficiently high structural "internal cohesive strength" to resist a 3 ft. head of water pressure. This is an important feature, for as will be appreciated it would not be of much value to fill a buried telephone cable with a composition of the type disclosed herein if that composition, at the ambient temperatures involved, could be extruded from the cable by dint of external water pressure applied following sheath damage.

The polybutene constituent of our compositions is poly(butene-1) as opposed to polyisobutylene. The average molecular weight of the polybutene we use is in the range of 500–3500, and desirably is in the range of 70–1100. A mixture of polybutenes may be used, as long as the average molecular weight is within the aforesaid range of 500–3500, and the product has the appropriate viscosity. Such polybutenes are viscous, oily liquids, and are prepared by the polymerization of n-butene in the presence of a halide olefin polymerizing catalyst, or by other methods well known to those skilled in the art.

Polybutenes which are suitable for use in the composition of this invention are those having a viscosity in the range of 850–1150 SUS/98.8°C.

One preferred polybutene for use in the present invention is one meeting the following specifications:

Specific Gravity/15.6/15.6°C.	0.890
Viscosity/37.8°C.	38,400 SUS
Colour APHA	10
Flash Point (C.O.C.)	204.4°C
Fire Point	232.2°C
Pour Point	–1.1°C
Total Chlorides ppm	5
Total Sulphur ppm	5
Water Content	Nil

Another preferred polybutene for use in the compositions of this invention is one having the following characteristics:

Average molecular weight 920

Viscosity SU/37.8°C. (ASTM D 2161) 35,944

Viscosity SUS/98.8°C (ASTM D 2161) 985

Viscosity index (ASTM D 567) 109

Evaporation loss (10 hr. at 98.8°C), wt. % (ASTM D972) 0.0

Pour Point, (ASTM D 97) —6.7°C.

Acidity, mg. KOH/gm (ASTM D974) 0.01

Specific Gravity (15.6°C) 0.882

Microcrystalline slackwax, one of the ingredients of our inventive composition, is a material of variable composition; however, normally such a material would have a mineral oil content of 15% – 40% and have a congealing point of 54.5° – 79.5°C, the waxes present being of microcrystalline structure. Microcrystalline waxes, as is well known, are composed of saturated hydrocarbon compounds of 40–50 carbon atoms with av-

erage molecular weights of 500 to 800. In contrast to the paraffinic waxes, microcrystalline waxes cannot be distilled at atmospheric pressure without some decomposition. The compounds are largely branched chain molecules with the branches occuring at random along the carbon chain. These waxes have a crystal structure much smaller than that of paraffin wax. The melting point of refined microcrystalline waxes is typically in the range of 62.8° -90.5°C.

As previously indicated, there may be used as a substitute for the microcrystalline slackwax constituent, a mixture of brightstock oil, or other mineral oil such as a neutral or pale oil having equivalent physical properties, with microcrystalline waxes.

Brightstock oils are high V.I. (viscosity index) mineral oils which meet the general specification for high viscosity mineral oils previously listed, namely:

Viscosity/98.8°C - 120 SUS — 220 SUS
Viscosity/37.8°C - 2000 SUS — 3500 SUS
Flash Point — at least 204.4°C
Specific Gravity/15.6°C - 0.875 - 0.925
Colour — ASTM 2 — ASTM 8
Boiling Range (at atmospheric pressure) — 260° - 704.5°C.

(The abbreviation SUS signifies Saybolt Universal seconds).

Other mineral oils besides brightstock oil, e.g. neutral and pale oils, may also be used in the aforesaid mixtures with microsrystalline waxes, provided they have physical chracteristics similar to those given above.

More particularly the present invention, in one aspect, resides in a composition suitable for use in filling telecommunications cables or the like, having the following formulation:

Polybutene, of average molecular weight 920	50.0% - 65.0%
Microcrystalline slackwax	25.0% - 45.0%
Polyethylene of average molecular weight in the range of 10,000 to 20,000	1.0% - 8.0%
Synthetic paraffinic wax	1.0% - 10.0%
Silica flour	0.50 - 8.0%

The percentages of ingredients given above are by weight, relative to the entire composition.

In another broad aspect, this invention resides in a process for preparing a composition suitable for use in filling telecommunications cables and the like, comprising the following steps, in sequence;

a. mixing together, with heating, the following ingredients:

i. polybutene, of average molecular weight in the range of from 500 to about 3,500;

ii. either (a) microcrystalline slackwax; or (b) an equivalent thereof, comprising a mixture of at least one microcrystalline wax with a high viscosity mineral oil meeting the following general specifications:

Viscosity/98.8°C. — 120 SUS — 220 SUS
Viscosity/37.8°C. — 2000 SUS — 3500 SUS
Flash Point — 204.4°C Minimum
Specific Gravity/15.6°C. — 0.875 — 0.925
Colour ASTM 2 — ASTM 8
Boiling Range (at atmospheric pressure) — 260° - 704.5°C.;

iii. polyethylene, of average molecular wieght in the range of 10,000 to 20,000; and

iv. at least one wax selected from the group consisting of (1) paraffin waxes having melting points in the range of 37.8° - 79.5°C., (2) synthetic Fischer-Tropsch type waxes having melting points in the range of 71.1° - 115°C., and (3) natural waxes selected from the group: Beeswax, Carnauba wax, Chinese insect wax, Japan wax, Myrtle wax and Spermaceti wax, until a homogeneous liquid mixture is obtained; and

b. adding finely divided silica in small increments to the mixture while subjecting the latter to high velocity, high shear mixing, until complete dispersion of said silica in the mixture is achieved and a completely uniform product is obtained, each increment of said finely divided silica being thoroughly dispersed in the mixture before a succeeding increment is added, the amount of said finely divided silica used ranging from about 0.5% to about 8.0% by weight of the composition, and said polybutene being employed in an amount greater than that of any other constituent in the composition.

The compositions of the present invention have the following physical characteristics:

Viscosity(Brookfield)/98.8°C (ASTM D2669) 150 - 400 c.p.s.
Cone Penetration/25°C (ASTM D937) 80 - 120-1/10 mm units
Drop Point (ASTM D127 Method) 85°-95°C
Dissipation Factor/100°C/60 Hz 0.005 Maximum
Volume Resistivity/100°C (ASTM D257) 1×10^{10} ohms. metre Minimum

Our compositions are also characterized by being thixotropic. The term "thixotropic" is used herein to denote the property of certain materials to change rapidly, on standing, from a liquid into a gel-like solid mass or body having sufficient cohesive strength to withstand distortion by gravitational force when suspended freely in an inverted receptable or on a coated object. The gel is also of such a nature that it can be fluidized by the application of mechanical agitation as by shaking, stirring, vibrating, and the like. The property of thixotropy as understood herein is thus characterized by a reversible isothermal sol $\rightleftharpoons$ gel transition.

As to the wax component in our composition there may be used any of the following: 1. Crude and refined paraffin waxes having melting points in the range of 37.8° to 79.5°C. 2. Crude and refined microcrystalline waxes having melting points in the range of 62.8° - 90.5°C. 3. Synthetic Fischer-Tropsch type waxes, both crude and refined, having melting points in the range of 71.1° - 115°C.

Certain vegetable, insect and animal waxes may also be used in our compositions in place of one or more of the above categories of waxes. Suitable vegetable, insect and animal waxes for this purpose are as follows:

Beeswax (Apis Mellifera)
Carnauba Wax (Corypha Cerifera)
Chinese Insect Wax (Coccus Cerifera)
Japan Wax (Rhus Succedaneum)
Myrtle Wax (Myrica Cerifera)
Spermaceti Wax (Physeter Macrocephalus)

Of the above animal, insect, vegetable waxes, the most useful are Beeswax, Carnauba Wax, Chinese Insect Wax and Spermaceti Wax; however, the other waxes listed will produce acceptable end products if used within the range we have specified for the wax content of our telephone cable filling compositions and after suitable refining processes.

Another component of our composition is polyethylene, of molecular weight in the range of 10,000 to 20,000. A preferred polyethylene for use in the compositions of this invenntion is one having a molecular weight of approximately 19,500. This substance, which is produced by either high pressure or low pressure polymerization of ethylene, is a white thermoplastic material readily obtainable from a variety of plastics manufacturers.

In the compositions of this invention the polybutene/microcrystalline slackwax/polyethylene/-synthetic paraffin wax blend produces a soft, rather viscous "petrolatum" type material which, of its own accord, is not completely satisfactory for the purpose being considered, i.e. for filling telecommunication cables. By dispersing the correct grade of finely divided silica (i.e. silica flour), under the appropriate conditions, a gel-like structure is formed, which structure is sufficiently stable to preclude the separation of any of the components during storage, filling and/or use. The silica flour, i.e. gelling agent, produces a gel such that although the gel structure is lost on liquefaction, it is regenerated on cooling. The polybutene/microcrystalline slackwax/polyethylene/synthetic wax blend may be considered as one major component, formulated to introduce inherent strength into the final gel structure, with the silica flour acting as the second major component, the gelling agent.

As previously stated the gelling agent used in the compositions of the present invention is finely divided silica. This material may also be considered as a filler in the composition. The filler used is variously known as: silica flour, fumed silica, pulverized silica, atomized silica or micronized silica. The porportion of filler to gel-like substance required depends (a) on the desired end, liquid viscosity characteristic, (b) on the degree of hardness desired and (c) on the character and particle size of the filler itself. The character of the filler includes the shape and surface properties of the individual particles; the character of the particles determines the end, liquid viscosity and hardness characteristics. Plateshaped or needle-like particles will reduce the viscosity of the jelly-like substance more than spheroid particles. The smaller the particle size the smaller the amount of filler required.

Such a composition has thixotropic properties: that is when it is caused to move or is agitated, for example by pumping, its viscosity is reduced. In the case of flat or needle-like particles this viscosity reduction is assisted by the alignment of the particles with the direction of motion. When the compound is substantially at rest the particles will be randomly directed, and thus assist in increasing the viscosity.

The particle size range of finely divided silica (silica flour) may vary very considerably; however, that used in the compositions of the present invention range anywhere frm 0.007 to 0.050 microns. Silica flours we have found quite satisfactory for use in the present invention are those designed by the trademark CAB-O-SIL. The following is a description of this particular material.

CAB-O-SIL is one of the purest silicas commercially available. On a dry basis, it is 99% silicon dioxide and is practically free frm contaminating metallic oxide. It contains no calcium, sodium or magnesium. CAB-O-SIL is so pure it meets the requirements of the FDA for use in foods in concentrations up to 2%.

Particles of CAB-O-SIL range in size from 70 to 500 angstroms or 0.007 to 0.050 microns. The physical appearance is that of a fluffy, snow white, super fine powder of extermely low bulk density. CAB-O-SIL particles are finer than those of the finest grades of rubber reinforcing carbon blacks. They are as fine as cigarette smoke.

When thoroughly dispersed and mixed with clear liquids such as mineral oil and turpentine, a transparent product is obtained. When dispersed and mixed with liquids such as alkyd vehicles, polyester resins, dioctyl phthalate and other plasticisers and varnishes, a translucent product is produced. The refractive index of 1.46 is close to that of many organic liquids and therefore dispersions are relatively transparent or translucent. For example, a dispersion of CAB-O-SIL in butyl alcohol results in a perfectly clear suspension. CAB-O-SIL is made by a vapour phase process. It is produced by the hydrolysis of silicon tetrachloride at 1100°C. This process produces a colloidal silica of exceptional purity. CAB-O-SIL, because it is produced at a high flame temperature, is generally classified as a "fumed" silica.

From the physical properties and surface characteristics stems the ability of CAB-O-SIL fumed silica to impart thickening and thixotropic control to liquids.

When CAB-O-SIL is dispersed in a liquid system, the chain-like formulations join each other an form a network type of structure. This reduces the ability of the liquid to flow and results in increased viscosity or thickening. Upon agitation or shear, the network structure breaks down and reforms after agitation stops. When a gel reverts to a liquid upon agitation and reforms as a gel when agitation stops, the liquid is commonly known as being thixotropic. When very small amounts of CAB-O-SIL are dispersed in a liquid system, there is a limited amount of hydrogen-bonding, because the chains are generally too far apart to bond in a closely knit formation. By increasing the concentration of CAB-O-SIL to a point where there is a sufficient number of CAB-O-SIL chains which have hydrogen-bonded to each other, the desired thickening or thixotropy can be obtained.

A particularly suitable silica flour for the purposes of the present invention is that known under the Trademark "Cabosil M5" which has a particle size of 0.012 microns. Other silica flours which have been found satisfactory are those known under the Trademarks "Syloid 224," "Syloid 308," "Gasil 23," "Tixosil 38A," "Zeosil 39" and "Cabosil M7." The use of the proper grade of finely divided silica, plus the correct incorporating techniques, are the key to the manufacture of an acceptable composition according to this invention.

The amount of finely divided silica employed may range from about 0.5% to about 8.0% by weight of the composition. Preferably, however, we employ an amount of finely divided silica ranging from about 0.5% to about 5.0% by weight of the composition. Still more preferably, the finely divided silica is used in an amount ranging from about 1.0% to about 3.0% by weight of the composition.

The ingredients of our telephone cable filling compositions are obtainable from various sources. For instance, microcrystalline slackwaxes, microcrystalline and paraffin waxes, which are by-products of petroleum refining, are readily obtainable from petroleum refineries. Brightstock oil, if used in our product, may

be obtained from similar sources. A synthetic paraffin wax we have found to be particularly useful in formulating our compositions is one obtained from SASOL Ltd. of South Africa and sold under the trademark "PARAFLINT V.I.," Other suitable waxes of this general type, known in the trade as "Fishcer-Tropsch" waxes, are available from BASF in Germany and from other sources.

As an optional ingredient there may be included up to about 1.0% by weight of an antioxidant. Any of the well known antioxidants for stabilizing organic substances and materials may be used; for instance, suitable antioxidants for use in the compositions of this invention are those disclosed in British Pat. Specification 1,117,771 of Union Carbide Corporation, published June 26, 1968, and in U.S. Pat. No. 3,156,728 of Orloff et al, granted Nov. 10, 1964; and also in the reference "Autoxidation and Antioxidants," Lundberg (1962), Interscience Publishers, Inc., New York.

If desired, there may be added to our compositions as an optional ingredient, minor amounts of "a tackiness" agent: this could be either a resinous material or a material such as high molecular weight polyisobutylene.

The cable filling compositions of the present invention are prepared according to the following procedure:

a. The polybutene, microcrystalline slackwax, polyethylene and second wax component (e.g. synthetic paraffinic wax) are blended together, and are heated to approximately 125°C and the batch stirred until the wax and polyethylene constituents are complete dissolved. If any antioxidant is to be included in the composition, it is added at this stage. The order of addition of the various components mentioned above is not particularly important.

b. The finely divided silica (e.g. silica flour) is then added in small increments and thoroughly dispersed with high speed, high shear stirring until complete uniformity has been achieved as determined by drop point and viscosity measurements.

As previously stated the key to making an acceptable cable filling composition according to this invention is the proper dispersion of the finely divided silica, as well as the use of the proper grade of finely divided silica.

As suitable equipment for effecting the high speed, high shear stirring there may be used any high speed stirrer known to those skilled in the art, as long as it can impart sufficient shear energy to the composition. For insntance, mixers such as the "Lightning Mixer" (trade mark), the "Cowles Dissolver" (trade mark), the "Twin Daysolver" (trade mark) and various types of turbine mixers such as described in pages 1210–1211 of Perry, "Chemical Engineers' Handbood" 3rd Ed. (1950), McGraw-Hill Book Co., New York, are suitable. Also suitable are high speed high shear dispersing devices such as are described in Chapter 6 of the book "Practical Emulsions," 3rd Edition, Vol. 1, Bennett et al. (1968), Chemical Publishing Company, Inc., New York.

A typical batch of approximately 5 tons of the composition according to this invention is produced in a 24 hours period; this includes also the period required for testing of the composition.

The following example represents a preferred composition and method of preparation in accordance with the present invention. The percentages given are by weight, relative to that of the whole composition.

Example

55.5% Polybutene — Average Molecular Weight — 920

35.0% Microcrystalline Slackwax

4.0% Polyethylene, Molecular Weight — 19,500

3.0% Synthetic Paraffin Wax, m.p. in range of 71.1° – 115°C.

2.5% Silica Flour

The polybutene, microcrystalline slackwax, polyethylene and synthetic paraffin wax are heated together and blended at a temperature of 125°C. (An antioxidant, if desired, is also incorporated into the composition at this stage.) The mixture is allowed to cool to 110°C. at which temperature the silica flour is added in small increments with high speed/high shear stirring, until a completely uniform and consistent viscosity product is obtained. It is vital that the silica flour be added in small increments and thoroughly dispersed before the next increment is added and that sufficient shear energy be imparted to the composition during this process.

The compositions of this invention may be used to fill telephone cables by means known in the art. Generally speaking, the composition is heated until molten although it may also be used in the plastic solid state, and is then applied to preformed, plastic insulated telephone wires, at one or more filling points during the cable's manufacture, the composition being maintained at such a temperature (a) as not to "set-off" on contact with the cold cable and (b) as not to exude from the interior of the cable after it has been filled. Arrangements such as disclosed, for example, in British Pat. Specification No. 1,120,011 published Jul. 17, 1968; British Pat. Specification No. 1,136,344 published Dec. 11, 1968; U.S. Pat. No. 3,607,487 Biskeborn et al granted Sept. 21, 1971, or British Pat. Specification No. 1,293,942 published Oct. 25, 1972, may be used for the application of our inventive filling compositions.

Our compositions were evaluated as follows:

(a) High Temperature Melting Point — ASTM D127 and ASTM D566. (The minimum desired temperature for compositions of the type with which this invention is concerned, is 80°C. as measured by ASTM D566 and 85°C. as measured by ASTM D127). (b) Syneresis — i.e. the tendency to separation of oil/polymers from a formulation. There is no standardized test that is known for determining this property; however, it has been found that compounds held at 20° – 30°C. below their ASTM D127 drop points for periods of 7–14 days will show signs of liquid separation, if this is a characteristic of the compound. (c) Plasticity at Low Temperature (–40°C. – /–40°F.) - A Company devised test which consists of placing a quantity of the composition in a suitable container — usually 750 grams of the composition in a 1000 ml metal container, and chilling to the required test temperature in 3 stages over a 6 our period and meauring the hardness of the material at the final temperature by means of Cone Penetration. Additionally thin layers of the composition (5–10 mils) may be coated onto thin metal plates (20–30 mil) and cooled to the required test temperature, at which temperature the plate/compound is flexed and signs of cracking/fissuring noted. Our compositions were test using both the above methods. (d) Void Formation - A

company devised test which is related to the Coefficient of Expansion of the composition as measured at different temperatures over a given temperature range, the specific expansion values obtained being plotted as a curve on linear graph paper. In general terms, the flatter the Coefficient of Expansion curve for a given composition produced during the test cycle, the more desirable is the composition for this particular end use; this is particularly so the closer the curve is to a continuous value of 0.0007℃ which is the average coefficient of expansion value for petroleum oils. The test method used to measure the compound's coefficient of expansion is a Company devised test in which a small sample (approximately 50 grams) of compound is cooled under applied pressure to the lower test temperature and is temperated at this temperature for 12 hours. The sample is then raised in temperature, at a controlled rate, the expansion undergone by the compound during the heating cycle being mechanically transmitted to a recording chart. (e) Dielectric Constant and Change in Dielectric Constant with Increase in Frequency - ASTM D150 In the case of telephone cable filling compositions it is desirable that there be a minimum change in the Dielectric constant of the filling composition with change in test frequency. The Dielectric Constant of air is 1. Ideally any filling compound should have this value for Dielectric Constant; however, most petroleum based compositions have Dielectric Constants in the range of 2.0 – 2.4. The cable filling compositions of this invention fall within this range at all measured frequency levels. (f) Compatibilty with Insulating Materials - a Company devised test. Plastic coated telephone cable wires are immersed in the compositions to be tested at a prescribed temperature for a prescribed period of time and are subsequently air aged after which air aging the physical changes which have occurred in the plastic insulation, as the result of its immersion in the compound, and subsequent air againg are noted. (g) Penetration/25° and 65℃ - ASTM D937 - It is vital that the compositions for use as telephone cable filling compounds have acceptable penetration values, not only at the standard test temperature of 25℃. but also at an elevated temperature. For this purpose a temperature of 65℃ was selected as being both a realistic and meaningful temperature. It is possible to formulate a composition having an acceptable penetration at 25℃. only to have this composition disintegrate at 65℃. The meeting of acceptable cone penetration values at both temperatures is of prime importance in formulating compositions of this general type. (h) Volume Resistivity/100℃. - ASTM D257 - This test relates to the freedom of the composition from moisture and polar contaminants which have an adverse effect on the performance of the finished telephone cable, particularly at high frequencies. Resistivity is also recommended as a test to determine when a cable filling composition has deteriorated beyond an acceptable point for continued in-plant usage. (i) Viscosity - Brookfield Viscosimeter Method - To produce compositions having an acceptable viscosity is a prime requirement. It is possible to produce compositions of such a viscous consistency that full filling of the cable interstices is not possible in the time period allowed in plant production. Formulating to an acceptable viscosity, and for the composition to have such a viscosity, is of critical importance in producing "fully filled" cables.

The following typical test data were obtained for the composition of the Example, previously described:

Viscosity (Brookfield) /98.8℃. (ASTM D2669)	250 cps
Drop Point (ASTM D127)	92℃
Plasticity/–40℃	No cracking observed
Dielectric Constant/100℃ (ASTM D150)	2.3
Volume Resistivity/100℃ (ASTM D257)	15×10^{10} ohms Meter
Cone Penetration/25℃ (ASTM D937)	100
Cone Penetration/65℃ (ASTM D937)	170
Dissipation Factor/100℃/60 Hz	0.0020

While certain embodiments of this invention have been particularly described herein, it is to be understood that the invention is not to be limited to specific embodiments, since for example variations in the ingredients of the compositions, and/or the proportions of ingredients, and/or processing conditions for the manufacture of these compositions, will be contemplated by those skilled in the art, without departing from the broadest aspects of the invention. It is therefore intended that the invention be limited only by the claims which follow.

What we claim is:

1. A composition suitable for use in filling telecommunication cables and the like, comprising:

1. 50.0% - 65.0% poly(butene-1), of average molecular weight in the range of 500 - 3500;
2. 25.0% - 45.0% microcrystalline slackwax; or of an equivalent of said microcrystalline slackwax, said equivalent consisting essentially of a mixture of at least one microcrystalline wax with a high viscosity mineral oil meeting the following general specification:

   Viscosity/98.8℃ — 120 SUS — 220 SUS
   Viscosity/37.8℃. — 2000 SUS — 3500 SUS
   Flash Point — 204.4℃ Minimum
   Specific Gravity/15.6℃ — 01875 – 0.925
   Colour ASTM 2 — ASTM 8
   Boiling Range (at atmospheric pressure) – 260°–704.5℃;
3. 1.0% – 8.0% polyethylene, of average molecular weight in the range of 10,000 to 20,000;
4. 1.0% – 10.0% of at least one wax selected from the group consisting of
   a. paraffin waxes having melting points in the range of 37.8° – 79.5℃., (b) synthetic Fischer-Tropsch type waxes having melting points in the range of 71.1° – 115℃., and (c) natural waxes selected from the group: Beeswax, Carnauba wax, Chinese insect wax, Japan wax, Myrtle wax and Spermaceti wax; and
5. from about 0.5% to about 8.0% finely divided silica, said percentages of ingredients being by weight, based on the entire composition.

2. A composition as defined in claim **1** wherein the finely divided silica is present in an amount ranging from about 0.5% to about 5.0% by weight of said composition.

3. A composition as defined in claim **2** wherein the finely divided silica is present in an amount ranging from about 1.0% to about 3.0% by weight of said composition.

4. A composition as defined in claim **1** wherein the wax constituent is a crude or refined paraffin wax having a melting point in the range of 37.8° – 79.5℃.

5. A composition as defined in claim 1 wherein the wax constituent is a crude or refined synthetic Fisher-Tropsch type wax having a melting point in the range of 71.1° - 115°C.

6. A composition as defined in claim 1 wherein the second-mentioned ingredient is microcrystalline slackwax.

7. A composition as recited in claim 1 wherein the second-mentioned ingredient is a mixture of at least one microcrystalline wax and brightstock oil, said brightstock oil meeting the following general specification:

Viscosity/98.8°C. — 120 SUS — 220 SUS
Viscosity/37.8°C — 2000 SUS — 3500 SUS
Flash Point — 204.4°C Minimum
Specific Gravity/15.6°C — 0.875 - 0.925
Colour ASTM 2 — ASTM 8
Boiling Range (at atmospheric pressure) — 260° — 704.5°C.

8. A composition as set forth in claim 1, said composition meeting the following specifications:

Viscosity (Brookfield) /98.8°C. (ASTM D2669)	150–400 cps
Cone Penetration/25°C (ASTM D937)	80–120 1/10 mm units
Drop Point (ASTM D127 method)	85–95°C
Dissipation Factor/100°C/60Hz	0.005 maximum
Volume Resistivity/100°C (ASTM D257)	1×10^{10} ohms meter minimum.

9. A composition as defined in claim 1 which includes also an antioxidant in an amount of up to about 1.0% by weight of said composition.

10. A composition according to claim 1, wherein the poly(butene-1) is of average molecular weight 920, and the wax constituent is a synthetic Fischer-Tropsch type wax.

11. A composition as in claim 1 wherein the silica flour has an average particle size in the range of 0.007 - 0.05 microns.

12. A composition as in claim 11 wherein the silica flour has an average particle size of 0.012 microns.

13. A composition as in claim 1 wherein the polyethylene has an average molecular weight of about 19,500.

14. A composition as defined in claim 1, having the following formulation:

Poly(butene-1) of average molecular weight 920	55.5%
Microcrystalline slackwax	35.0%
Polyethylene, of average molecular weight 19,500	4.0%
Synthetic Paraffinic wax	3.0%
Silica Flour	2.5%,

said percentages of ingredients being by weight based on said composition.

15. A composition as in claim 1 wherein the poly(butene-1) constituent comprises a mixture of low molecular weight poly(butenes-1) of varying molecular weights and viscosities, said mixture having an average molecular weight within the range of 500 to about 3500.

16. A composition according to claim 1 wherein the poly(butene-1) constituent is one which meets the following specifications:

Specific Gravity/15.6/15.6°C.	0.890
Viscosity/37.8°C	38,400 SUS
colour APHA	10
Flash Point (C.O.C.)	204.4°C.
Fire Point	232.2°C.
Pour Point	−1.1°C
Total Chlorides ppm	5
Total Sulphur ppm	5
Water Content	Nil.

17. A composition according to claim 1 wherein the poly(butene-1) constituent is one which meets the following specifications:

Average molecular weight	920
Viscosity SUS/100°F. (ASTM D 2161)	35,944
Viscosity SUS/210°F. (ASTM D2161)	985
Viscosity index (ASTM D567)	109
Evaporation loss (10 hr. at 98.8°C.) weight percent (ASTM D972)	0.0
Pour Point (ASTM D 97)	−6.7°C.
Acidity, mg. KOH/gm (ASTM D 974)	0.01
Specific Gravity (15.6°C.)	0.882.

18. Process of preparing a composition suitable for use in filling telecommunication cables and the like, comprising the following steps, in sequence:

a. mixing together, with heating, the following ingredients:

i. poly(butene-1) of average molecular weight in the range of from 500 to 3500;

ii. microcrystalline slackwax; or an equivalent of said microcrystalline slackwax, consisting essentially of a mixture of at least one microcrystalline wax with a high viscosity mineral oil meeting the following general specification:

Viscosity/98.8°C. — 120 SUS — 220 SUS
Viscosity/37.8°C. — 2000 SUS — 3500 SUS
Flash Point — 204.4°C. Minimum
Specific Gravity/15.6°C. — 0.875 - 0.925
Colour ASTM 2 — ASTM 8
Boiling Range (at atmospheric pressure) — 260° - 704.5°C;

iii. polyethylene, of average molecular weight in the range of 10,000 to 20,000; and

iv. at least one wax selected from the group consisting of (1) paraffin waxes having melting points in the range of 37.8° - 79.5°C., (2) synthetic Fischer-Tropsch type waxes having melting points in the range of 71.1° - 115°C., and (3) natural waxes selected from the group: Beeswax, Carnauba wax, Chinese insect wax, Japan wax, Myrtel wax and Spermaceti wax, until a homogeneous, liquid Myrtle is obtained; said ingredients being employed in the following relative proportions:

Poly(butene-1)	50.0% - 65.0%
Ingredient (ii)	25.0% - 45.0%
Polyethylene	1.0% - 8.0%
Ingredient (iv)	1.0% - 10.0%,

said percentages of ingredients being by weight, based on the entire composition;

b. adding finely divided silica in small increments to the mixture while subjecting the latter to high velocity, high shear mixing until complete dispersion of said silica in the mixture is achieved and a completely uniform product is obtained, each increment of said finely divided silica being thoroughly

dispersed in the mixture before a succeeding increment is added, the amount of said finely divided silica used ranging from about 0.5% to about 8.0% by weight of the composition.

19. A process as in claim **18,** wherein in step (a) the second-mentioned ingredient is microcrystalline slackwax.

20. A process as in claim **18** wherein the amount of finely divided silica used is in the range of about 0.5% to about 5.0% by weight of the composition.

21. A process as in claim **18** wherein the amount of finely divided silica used is in the range of about 1.0% to about 3.0% by weight of the composition.

22. A process as in claim **19** wherein the wax constituent is a synthetic Fisher-Tropsch type wax having a melting point in the range of 71.1° – 115°C.

23. A process as in claim **22** wherein, in step (a), the poly(butene-1), microcrystalline slackwax, polyethylene and synthetic wax are heated together at a temperature of approximately 120°C. until a homogeneous mixture is obtained.

24. A process as set forth in claim **23** wherein, after said homogeneous mixture is obtained, it is cooled to about 110°C. at which temperature step (b) is carried out.

25. A process as defined in claim **18,** wherein there is included also an antioxidant, in an amount up to about 1.0% by weight of the composition, said antioxidant being incorporated into the composition together with said poly(butene-1), microcrystalline slackwax or equivalent thereof, polyethylene and wax, in the first step.

26. A process as defined in claim **18,** wherein the poly(butene-1) is of average molecular weight 920, and the wax constituent of the composition is a synthetic Fischer-Tropsch type wax.

27. A process as in claim **18** wherein the silica flour has an average particle size in the range of 0.007 – 0.05 microns.

28. A process as in claim **27** wherein the silica flour has an average particle size of 0.012 microns.

29. A process as in claim **18** wherein the polyethylene has an average molecular weight of about 19,500.

30. A process as in claim **18** wherein the ingredients used and their relative proportions, are as follows:

Poly(butene-1), of average molecular weight 920	55.5%
Microcrystalline slackwax	35.0%
Polyethylene, of average molecular weight 19,500	4.0%
Synthetic paraffinic wax	3.0%
Silica flour	2.5%,

said percentages of ingredients being by weight, based on the composition.

31. A process according to claim **18** wherein the poly(butene-1) constituent is one which meets the following specifications:

Specific Gravity/15.6/15.6°C.	0.890
Viscosity/37.8°C.	38,400 SUS
Color APHA	10
Flash Point (C.O.C.)	204.4°C.
Fire Point	232.2°C.
Pour Point	−1.1°C.
Total Chlorides ppm	5
Total Sulphur ppm	5
Water Content	Nil.

32. A process according to claim **18** wherein the poly(butene-1) constituent is one which meets the following specifications:

Average molecular weight	920
Viscosity SUS/100°F. (ASTM D2161)	35,944
Viscosity SUS/210°F. (ASTM D2161)	985
Viscosity index (ASTM D 567)	109
Evaporation loss (10 hr. at 98.8°C) weight percent (ASTM D972)	0.0
Pour Point (ASTM D97)	−6.7°C
Acidity, mg. KOH/gm. (ASTM D974)	0.01
Specific gravity (15.6°C.)	0.882.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,893,962

DATED : July 8, 1975

INVENTOR(S) : Basil Vivian Edwin Walton
William Edward John Wannamaker

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Abstract, line 1, delete "if" and substitute --is--.
Col. 3, line 47, delete "te" and substitute --the--;
line 47, delete "commprising" and substitute --comprising--.
Col. 4, line 26, delete "70-1100" and substitute --700-1100--;
line 53, delete "SU" and substitute --SUS--.
Col. 7, line 4, delete "invenntion" and substitute --invention--.
line 57, delete "frm" and substitute --from--;
line 64, delete "frm" and substitute --from--.
Col. 8, line 28, delete "an" and substitute --and--.
Col. 9, line 21, delete ""a tacki-" and substitute --a "tacki- --
line 33, delete "complete" and substitute --completely--;
line 55, delete "Handbood" and substitute --Handbook--;
line 64, delete "hours" and substitute --hour--.
Col. 10, line 59, delete "our" and substitute --hour--;
line 66, delete "test" and substitute --tested--.
Col. 12, line 39, delete "01875" and substitute --0.875--.

Add to the listed Foreign Patents or Applications the following
932,885 8/1973 Canada

Signed and Sealed this

Twentieth Day of July 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks