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(54) **COMPONENT FOR LUBRICANTS**

(57) Oligomeric or polymeric compound based on amino acids and/or their derivatives, preferably for application in lubricants, e.g. as thickeners, as well as a process for the production of a component of a lubricant, preferably a thickener, comprising an oligomeric or polymeric compound based on amino acids and/or their derivatives

and use of an oligomeric or polymeric compound based on amino acids and/or their derivatives for the production of (a component of) lubricants (lubricant greases, lubricating oils, and water-based lubricants). In particular, the production of (multifunctional) lubricating greases.

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Description

[0001] The invention relates to the field of lubricants (lubricating greases, lubricating oils and water-based lubricants), more particular to components or additives for lubricants such as lubricating agents and/or thickeners for lubricants, especially for lubricating greases.

[0002] Lubricants primarily serve to reduce friction between elements moving relative to each other. Depending on their specific composition, in particular their additives, they can have also various other functions, such as increasing the wear or tear resistance or confer corrosion protection.

[0003] Lubricants are compositions that typically comprise at least one lubricating agent, at least one thickener and optionally additives. Most often, the manufacture of these components is based on petrochemical raw materials.

[0004] Accordingly, it is an objective of the present invention to provide a component for a lubricant that is biobased and a biobased way of production thereof. This means that the raw material is non-fossil and the process of manufacture does not necessarily involve a petrochemical step. Ideally, this component is dual or multifunctional, i.e. it has at least two or more functional properties, for example it is a thickener and serves also to reduce the wear resistance.

[0005] This objective is solved by an oligomeric or polymeric compound based on amino acids and/or their derivatives according to claim 1 and a process for its production. This compound is particularly suitable for providing lubricants, in particular lubricating greases.

[0006] According to the invention amino acids or their derivatives are employed as a basis for the production of oligomeric or polymeric compounds, preferably as component e.g. thickener or additive for lubricants. The amino acids can be polymerized with (di)isocyanates, diols, diamines or dithiols, as well as compounds with combinations of these functional groups. To achieve a better thickening effect, the carboxylic acid function of the amino acids can be chemically modified.

[0007] The oligomeric or polymeric compound according to the invention can serve as a multifunctional thickener with additional functions (oxidation stability, corrosion protection, EP-AW). This can make the use of additives obsolete.

[0008] The oligomeric or polymeric compound, preferably for lubricants according to the invention may be produced using mechanochemical processes, such as ball milling that allow for lower reaction temperatures and shorter reaction times. By using a ball mill for example, it is possible to produce at 50-60°C and with reaction times of 30-45 minutes. In addition, the ball mill process can also be used as a continuous process. This makes it possible to save resources (energy) and also process temperature-sensitive additives can be processed.

[0009] In addition, disadvantages of high temperature control, such as unpleasant odors or fogging are avoided, which are typically a problem with lubricating greases.

[0010] In brief, the invention has one or more of the following advantages:

- sustainable/renewable (for natural amino acids).
- multifunctional (for amino acids with additional functional groups)
- versatile modifiable (through modification of the carboxylic acid group by modification of further functional groups)
- energy-saving production with mechanochemical processes.

[0011] The oligomeric or polymeric compound, preferably for application in lubricants, consists of at least one monomeric unit X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and two units Y, derived from an organic compound [Y] that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group and that is no carboxyl group or a derivative thereof, and optionally at least one monomeric unit A, derived from a monomer [A] featuring two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomers [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxamide group and wherein [Y] has a primary amine group and additionally at least one carboxamide group, if the oligomeric or polymeric compound does not contain A.

[0012] In a preferred embodiment, the component, preferably for lubricants is an oligomeric or polymeric compound consisting of at least one monomeric unit X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and two units Y, derived from an organic compound [Y] that has one isocyanate group or one primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group, wherein a) [Y] has a primary amine group and additionally at least one carboxyl group or a derivative thereof, preferably a carboxamide group, or b) the oligomeric or polymeric compound additionally consists of at least one monomeric unit A, derived from a monomer [A] that has two functional groups, wherein the functional groups are the same or different and selected from primary or

secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxamide group.

[0013] "Amino acids" according to this invention are chemical compounds with a nitrogen (N) containing amino group and a carboxylic acid group containing carbon (C) and oxygen (O). In "natural amino acids", only one hydrocarbon moiety is present between the amino group and the carboxylic acid group. In the case of artificial amino acids, the length of the hydrocarbon moiety may vary. In addition to amine and acid functionality, naturally occurring amino acids may feature a number of different functional groups (S-CH₃, -OH, -NH₂, -COOH etc.), which are relevant for the performance of a lubricating grease (e.g. friction reduction, wear protection, corrosion protection). Examples for amino acids are: histidine, lysine, methionine, phenylalanine, threonine, tryptophan, asparagine, aspartic acid/aspartate, alanine, arginine, cysteine/cystine, glutamine, glutamic acid/glutamate, glycine, proline, serine, tyrosine, hydroxyproline/oxoproline and ornithine.

[0014] An "oligomer" according to the invention is a molecule of intermediate relative molecular mass, the structure of which essentially comprises a small plurality of units (monomeric units) derived, actually or conceptually, from molecules of lower relative molecular mass (monomers). A molecule is regarded as having an intermediate relative molecular mass, if it has properties which do vary significantly with the removal of one or a few of the units. If a part or the whole of the molecule has an intermediate relative molecular mass and essentially comprises a small plurality of units derived, actually or conceptually, from molecules of lower relative molecular mass, it may be described as "oligomeric". Oligomers are usually named according to the number of units such as dimer, trimer, tetramer, pentamer, hexamer, heptamer, octamer.

[0015] A "polymer" according to the invention is a macromolecule, a molecule of high relative molecular mass, the structure of which essentially comprises the multiple repetition of units (monomeric units) derived, actually or conceptually, from molecules of low relative molecular mass (monomers). In many cases, especially for synthetic polymers, a molecule can be regarded as having a high relative molecular mass, if the addition or removal of one or a few of the units has a negligible effect on the molecular properties. If a part or the whole of the molecule has a high relative molecular mass and essentially comprises the multiple repetition of units derived, actually or conceptually, from molecules of low relative molecular mass, it may be described as either macromolecular or polymeric.

[0016] The term "derived from" according to the invention relates to the monomeric unit as present in the oligomer or polymer, that derives from the former monomer and deviates from the monomer due to the bonds formed by polymerization of the monomers to an oligomeric or polymeric compound.

[0017] The term "radical" according to this invention does not relate to radical polymerization but defines a moiety, meaning a part of a molecule that is bonded by a covalent bond to the molecule. Hence, the term radical throughout the application is interchangeable with the term moiety.

[0018] A "hydrocarbon" according to the invention is a compound consisting of carbon and hydrogen. According to IUPAC nomenclature, aliphatic compounds are "acyclic or cyclic, saturated or unsaturated hydrocarbon compounds, other than aromatic compounds".

[0019] The term "divalent" is referring to a molecule or atom, that is capable of forming chemical bonds with two other atoms e.g. of other molecules.

[0020] Monofunctional or bifunctional according to the present invention is referring to the presence of one or two of the respective functional groups in a molecule, respectively.

[0021] "Diisocyanates" are molecules that have two isocyanate groups of the formula (-NCO), wherein N is nitrogen, C is carbon and O is oxygen.

[0022] An "organic compound" according to this invention is no inorganic compound, meaning a chemical compound that comprises carbon-hydrogen or carbon-carbon bonds.

[0023] The term "functional group" according to this invention refers to a substituent or moiety in a molecule that causes the molecule's characteristic chemical reactions. The same functional group will undergo the same or similar chemical reactions regardless of the rest of the molecule's composition.

[0024] An "amine" according to this invention is a functional group or a compound comprising a functional group that contains a basic nitrogen atom with a lone pair. Amines are formally derivatives of ammonia (NH₃), wherein one (primary amine), two (secondary amine) or three (tertiary amine) hydrogen atoms have been replaced by a substituent such as an alkyl or aryl group.

[0025] A "sulfanyl" according to this invention is also known as mercapto radical, hydro-sulfide radical, or hydrosulfur and is the functional group of thiols consisting of one hydrogen and one sulfur atom (-SH).

[0026] A "hydroxyl" according to this invention is the functional group of alcohols with the chemical formula (-OH) and composed of one oxygen atom covalently bonded to one hydrogen atom.

[0027] A "carboxyl group" according to this invention is the functional group (-C(=O)OH).

[0028] A "carboxamide group" according to this invention is a functional group with the general formula -C(=O)NR'R", where R' and R" represent hydrocarbon radicals or hydrogen atoms, preferably R' is a hydrocarbon radical and R" is a

hydrogen atom.

[0029] "Carboxylate esters" are molecules having a functional group or a functional group according to formula $(-C(=O)OR)$.

[0030] "Thioesters" are compounds with the functional group $(-S-C(=O)-R)$. They are analogous to carboxylate esters with the sulfur in the thioester playing the role of the linking oxygen in the carboxylate ester. They are the product of esterification between a carboxylic acid and a thiol.

[0031] "Hydrophobic amino acids" according to this invention are amino acids that have hydrophobic side chains such as glycine (Gly), alanine (Ala), valine (Val), leucine (Leu), isoleucine (Ile), proline (Pro), phenylalanine (Phe), methionine (Met), and tryptophan (Trp). These side chains are composed mostly of carbon and hydrogen, have very small dipole moments, and tend to be repelled from water.

[0032] According to this invention O means oxygen, S is sulfur, N is nitrogen and C is carbon.

[0033] A "ball mill" is a type of grinder used to grind or blend materials. A ball mill consists of a hollow cylindrical shell rotating about its axis. The axis of the shell may be either horizontal or at a small angle to the horizontal. It is partially filled with balls. The grinding media are the balls, which may be made of steel (chrome steel), stainless steel, ceramic, or rubber. The inner surface of the cylindrical shell is usually lined with an abrasion-resistant material such as manganese steel or rubber lining. Less wear takes place in rubber lined mills. The length of the mill is approximately equal to its diameter.

[0034] "Functional groups, that react per addition reaction with isocyanate groups" can also be called "functional groups reactive to isocyanate" and comprise functional groups such as e.g. primary or secondary amine group, a sulfanyl group or hydroxyl group. Isocyanate reactions mainly occur through addition to C=N double bond. An active hydrogen atom-containing nucleophilic center attacks the electrophilic carbon atom and active hydrogen is added to the nitrogen atom.

[0035] X is a monomeric unit derived from an isocyanate with at least two isocyanate groups, preferably a diisocyanate or polymethylene polyphenylisocyanate (also known as polymeric MDI, p-MDI or PMDI), more preferably a diisocyanate.

[0036] Preferably X is a monomeric unit according to the formula $-[OCNH-R_2-NHCO]-$, wherein R_2 is a divalent hydrocarbon radical. The monomeric unit derives from a diisocyanate [X] according to the formula $OCN-R_2-NCO$, wherein R_2 is a divalent hydrocarbon radical, that reacted per addition reaction, preferably with a functional group selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group.

[0037] Suitable diisocyanates may be aliphatic or aromatic, cyclic, linear or branched.

[0038] Examples for suitable diisocyanates are 1,6-hexamethylene diisocyanate (HDI), 2-methylpentamethylene 1,5-diisocyanate, pentamethylene 1,5-diisocyanate (PDI), optionally provided as trimer, optionally in solution, 2,2,4- and 2,4,4-trimethyl-1,6-hexamethylene diisocyanate (TMDI), 1,12-dodecamethylene diisocyanate, lysine and lysine ester diisocyanate, cyclohexane 1,3-diisocyanate, cyclohexane 1,4-diisocyanate, 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane (isophorone diisocyanate or IPDI), perhydro-2,4'-diphenylmethane diisocyanate and perhydro-4,4'-diphenylmethane diisocyanate, 1,4-diisocyanato-2,2,6-trimethyl-cyclohexane (TMCDI), 1,3- and 1,4-bis(isocyanatomethyl)-cyclohexane, m- and p-xylylene diisocyanate (m- and p-XDI), m- and p-tetramethyl-1,3-xylylene diisocyanate, m- and p-tetramethyl-1,4-xylylene diisocyanate, bis(1-isocyanato-1-methylethyl)-naphthalene, 2,4- and 2,6-tolylene diisocyanate (TDI), 4,4', 2,4', and 2,2'-diphenylmethane diisocyanate (MDI), a mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate (MPI), 1,3- and 1,4-phenylene diisocyanate, 2,3,5,6-tetramethyl-1,4-diisocyanatobenzene, naphthalene 1,5-diisocyanate (NDI), 3,3'-dimethyl-4,4'-diisocyanatobiphenyl (TODD, oligomers and polymers of the aforesaid isocyanates, and mixtures thereof. However, this list shall not limit the invention by any means.

[0039] In one embodiment, the diisocyanate [X] is selected to be an aromatic diisocyanate and is preferably selected from 4,4', 2,4', and 2,2'-diphenylmethane diisocyanate (MDI), a mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate (MPI) or toluene diisocyanate (TDI), or mixtures thereof.

[0040] In another embodiment the diisocyanate [X] is selected to be an aliphatic diisocyanate such as hexamethylene diisocyanate (HDI), methylene dicyclohexyl diisocyanate or hydrogenated MDI (HMDI) and 1-isocyanato-3,3,5-trimethyl-5-isocyanatomethylcyclohexane (isophorone diisocyanate or IPDI) or mixtures thereof, preferably a linear or branched aliphatic diisocyanate, more preferably pentamethylene 1,5-diisocyanate (PDI). Preferably the diisocyanate [X] is selected to be an aliphatic diisocyanate according to the formula $OCN-R_2-NCO$, wherein R_2 is a divalent C2 to C20 alkyl, preferably with an odd number of carbon atoms, preferably C4 to C9. More preferably R_2 is a divalent, linear C5, C7 or C9 alkyl, most preferably C5 alkyl.

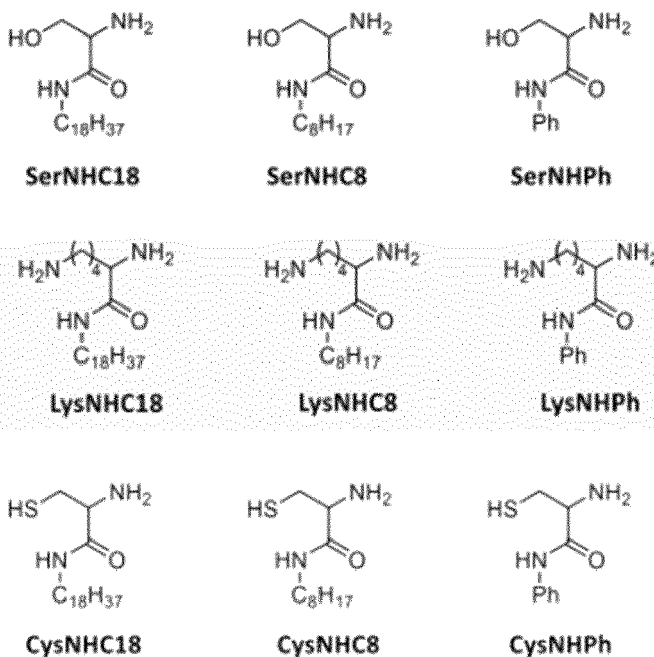
[0041] In a preferred embodiment A is a monomeric unit according to the formula $-[Z_1-R_1-Z_1]-$ wherein Z_1 is the same or different and is selected from O, S, N or NH, and wherein R_1 is a divalent hydrocarbon radical optionally having a carboxamide group.

[0042] Derivatives of amino acids can be employed and especially for polar amino acids this derivatization is of great advantage. Derivatives of amino acids were prepared, and it was found that these derivatives are particularly excellent for lubricant production and relatively low quantities are necessary for adequate gel formation. In addition, derivatization allows further functionalities can be incorporated into the lubricant or thickener structure.

[0043] In an especially preferred embodiment at least a part of the monomeric units A derives from amino acid deriv-

atives, preferably amino acid amides, that have a carboxamide group, preferably instead of the carbonyl group.

[0044] Exemplary structures for amino acid amides (derivatives of amino acids) such as serine, lysine and cysteine are the following:



[0045] In a preferred embodiment the two functional groups, that react per addition reaction with isocyanate groups, that are no carboxyl groups or derivatives thereof, of monomer [A] are not bound to the at least one carboxyl group or derivatives thereof, preferably the at least one carboxamide group.

[0046] In an especially preferred embodiment at least a part of monomers [A], preferably monomers [A], are amino acid amides, more preferably amino acid amides deriving from natural amino acids, preferably selected from tyrosine, serine, threonine, cysteine, selenocysteine, asparagine, glutamine, aspartate, glutamate, lysine, arginine, histidine, or mixtures thereof, more preferably lysine, cysteine and/or serine.

[0047] In one embodiment the oligomeric (or polymeric) compound is preferably a trimer and consists of one monomeric unit X, derived from a diisocyanate monomer [X], according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and two units Y, derived from an organic compound [Y] that has one primary amine group and one carboxamide group.

[0048] In this embodiment Y preferably derives from a modified amino acid, such as 12-amino stearic acid, preferably a modified natural amino acid [Y], more preferably amino acid amides deriving from natural amino acids, preferably selected from alanine, valine, leucine, isoleucine, methionine, proline, tryptophane, phenylalanine, more preferably alanine wherein the one functional group that reacts per addition reaction with an isocyanate is a primary amine group and wherein the carboxy group has been modified by amidation into a carboxamide group, preferably with the formula $-\text{C}(=\text{O})\text{NR}'\text{R}''$, wherein R' and R'' represent hydrocarbon radicals or hydrogen atoms, preferably R' is a hydrocarbon radical and R'' is a hydrogen atom.

[0049] R' can be linear, branched or cyclic. R' preferably is a hydrocarbon radical with C1 - C20, more preferably C6-C18. In one embodiment R' is a linear hydrocarbon radical with C8-C18, more preferably C8 or C18, preferably according to the formulas C_8H_{17} or $\text{C}_{18}\text{H}_{37}$, respectively. Alternatively, R' is a phenyl moiety according to the formula C_6H_5 .

[0050] In this embodiment [Y] is most preferably selected from 12-amino-octadecyl carboxamide or 2-aminopropanoic carboxamide.

[0051] Thus, in an especially preferred embodiment, the oligomeric or polymeric compound, preferably for application in lubricants, consists of at least one monomeric unit X, derived from PDI or MDI, and two units Y, derived from an organic compound that has a primary amine group and one carboxamide group, preferably a derivative of an amino acid such as 12-amino stearic acid, more preferably of a natural amino acid, especially alanine.

[0052] In an alternative embodiment the oligomeric or polymeric compound consists of at least two monomeric units X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate monomer according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and two units Y, derived from an organic compound [Y] that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate, and that is no carboxyl group or a derivative thereof, and at least one monomeric unit A, derived from a

monomer [A] that has two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the two functional groups are the same or different and wherein at least a part of the monomers [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxamide group.

[0053] In a preferred embodiment the oligomeric or polymeric compound consists of at least two monomeric units X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate monomer according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and two units Y, derived from an organic compound [Y] that has one isocyanate group or one functional group selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a secondary amine group, and at least one monomeric unit A, derived from a monomer [A] that has two functional groups, wherein the two functional groups are the same or different and selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomers [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxamide group.

[0054] In this embodiment Y is preferably an organic unit according to the formula $[\text{R}_3\text{-Z}_3]\text{-}$, wherein $\text{Z}_3 = \text{O}, \text{S}, \text{N}, \text{NH}$ or NHCO and wherein R_3 is a monovalent hydrocarbon radical. R_3 preferably is a monovalent hydrocarbon radical, preferably a C2 to C20 alkyl, more preferably C6 to C12. Y is preferably derived from monofunctional amines, alcohols, thioles or isocyanates [Y].

[0055] Preferably [Y] is a monofunctional amine, such as octyldiamine or stearylamine, preferably a biobased monofunctional amine, such as stearylamine.

[0056] In this embodiment optionally, a part of monomers [A] is selected from diamines, bifunctional thioles or dioles, preferably aliphatic diamines, bifunctional thioles or dioles, preferably that are no amino acids, more preferably according to the formula $[\text{Z}_1\text{-R}_1\text{-Z}_1]\text{-}$ wherein R_1 is a divalent hydrocarbon radical, preferably a C2 to C20 alkyl, more preferably C4 to C10. Diamines are organic compounds that have two amine groups, such as 1,6-diaminohexane or 1,5-diaminopentane. Bifunctional thioles are compounds that have two thiol groups such as propane-1,3-dithiol. Dioles are organic compounds that have two hydroxyl groups, such as ethylene glycol.

[0057] In an especially preferred embodiment a part of [A] is a diamine based on furane, preferably a furan-containing diamine, such as 2,5-furan diamine, 2,5-tetrahydrofuran diamine or propane-2,2-diylbis(furan-5,2-diyl)dimethanamine (PDFA), that can be derived from furfurylamine, more preferably a renewable diamine based on furan, such as propane-2,2-diylbis(furan-5,2-diyl)dimethanamine (PDFA).

[0058] In a preferred embodiment the oligomeric or polymeric compound consists of at least two monomeric units X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate monomer according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and two units Y, derived from an organic compound [Y] that has one isocyanate group or one functional group selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a secondary amine group, and at least one monomeric unit A, derived from a monomer [A] that has two functional groups that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the two functional groups are the same or different and selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer(s) [A], preferably the monomer(s) [A] is an amino acid amide.

[0059] In an especially preferred embodiment the oligomeric or polymeric compound consists of at least two monomeric units X, derived from an diisocyanate, preferably an aliphatic diisocyanate, more preferably PDI, and two units Y, derived from a monoamine, preferably stearylamine or a monofunctional isocyanate, preferably stearylisocyanate, and at least one monomeric unit A, derived from a monomer [A] that has two functional groups that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the two functional groups are the same or different and selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer(s) [A], preferably the monomer(s) [A] has two functional groups that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein one group is a primary amine group, and one carboxamide group, preferably the monomer(s) [A] derive from amino acids.

[0060] In an especially preferred embodiment, the oligomeric or polymeric compound has the general formula (I) or (II), more preferably (I), wherein n preferably is an integer from 1 to 20, preferably from 2 to 15, more preferably from 5 to 10, especially preferred 9.



[0061] The oligomeric or polymeric compound with Y according to the formula $[\text{R}_3\text{-Z}_3]\text{-}$, wherein R_3 is a monovalent

hydrocarbon radical has the general formula (I) when $Z_3 = O, S, N, NH$. The oligomeric or polymeric compound with Y according to the formula $[R_3-Z_3]_n$, wherein R_3 is a monovalent hydrocarbon radical has the general formula (II) when $Z_3 = NHCO$.

[0062] The oligomeric or polymeric compound has the general formula (I) when Y derives from a monoamine, an alcohol or a monofunctional thiol. The oligomeric or polymeric compound has the general formula (II) when Y derives from a (monofunctional) isocyanate.

[0063] The invention further relates to a process for production of a component of a lubricant, preferably of a lubricant, more preferably comprising an oligomeric or polymeric compound according to the invention, by contacting component [Y], wherein [Y] is an organic compound that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group, that is no carboxyl group or a derivative thereof, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group, and optionally monomer [A], wherein [A] has two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxyl group or a derivative thereof, preferably a functional group selected from a carboxyl group, carboxylate ester, thioester group or carboxamide, and wherein [Y] has a primary amine group and additionally at least one carboxyl group or a derivative thereof, if no monomer [A] is added, with component [X], wherein [X] is at least one isocyanate monomer with at least two isocyanate groups, preferably a diisocyanate monomer according to the formula $OCN-R_2-NCO$, wherein R_2 is a divalent, hydrocarbon radical, characterized in that the components are contacted in the presence of oil and/or wherein a mechanochemical, ultrasonication and/or sonochemical method is applied. In an especially preferred process for production of a component of a lubricant, preferably of a lubricant, more preferably comprising an oligomeric or polymeric compound according to the invention, is taken out by contacting component [Y], wherein [Y] is an organic compound that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group, that is no carboxyl group or a derivative thereof, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group and wherein [Y] has a primary amine group and additionally at least one carboxamide group with component [X], wherein [X] is at least one isocyanate monomer with at least two isocyanate groups, preferably a diisocyanate according to the formula $OCN-R_2-NCO$, wherein R_2 is a divalent, hydrocarbon radical, characterized in that the components are contacted in the presence of oil and/or wherein a mechanochemical, ultrasonication and/or sonochemical method is applied.

[0064] In this embodiment [Y], is an amino acid or a derivative thereof, preferably a natural amino acid, such as alanine, or a derivative thereof with a primary amine group and one carboxyl group or a derivative thereof and [X], wherein [X] is p-MDI or at least one diisocyanate monomer selected from PDI, MDI or IPDI, preferably at least one diisocyanate monomer selected from PDI, MDI or IPDI.

[0065] The chain growth of the polymerization is terminated with an endcapper [Y], such as a monoamine or a mono-isocyanate, so that the chain length is dependent on the ratio of [A] and [Y] or [X] and Y. The chain length of the thickeners has a great influence on the properties of the final products. Thus, in addition to the pure chemistry of [A], the properties of the product can also be influenced by the chain length of the product and the end group used.

[0066] In an alternative embodiment the process for production of a component of a lubricant, preferably of a lubricant, more preferably comprising an oligomeric or polymeric compound according to the invention, is taken out by contacting components [Y] and [A], wherein [Y] is an organic compound that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group, that is no carboxyl group or a derivative thereof, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group, and wherein monomer [A] has two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxyl group or a derivative thereof, preferably a functional group selected from a carboxyl group, carboxylate ester, thioester group or carboxamide, with component [X], wherein [X] is at least one isocyanate monomer with at least one isocyanate group, preferably a diisocyanate monomer according to the formula $OCN-R_2-NCO$, wherein R_2 is a divalent hydrocarbon radical, characterized in that the components are contacted in the presence of oil and/or wherein a mechanochemical, ultrasonication and/or sonochemical method is applied.

[0067] In an especially preferred embodiment at least a part of monomers [A] in the process have at least one functional group selected from a carboxylate ester, thioester group or carboxamide, preferably a carboxamide, according to formula (III):



wherein Z is selected from O, NH or S, preferably NH, and R is a hydrocarbon radical, preferably an aliphatic linear hydrocarbon radical. R can be linear, branched or cyclic. R preferably is a hydrocarbon radical with C1 - C20, more preferably C6-C18. In one embodiment R is a linear hydrocarbon radical with C8-C18, more preferably C8 or C18, preferably according to the formulas C₈H₁₇ or C₁₈H₃₇, respectively. Alternatively, R is a phenyl moiety according to the formula C₆H₅.

[0068] In an especially preferred embodiment at least a part of monomers [A], preferably monomers [A], are amino acids or derivatives thereof, such as amino acid amides, esters or thioesters, more preferably amino acid amides.

[0069] The amino acid derivatives preferably derive from natural amino acids.

[0070] In this embodiment the process for production of a component of a lubricant, preferably of a lubricant, more preferably comprising an oligomeric or polymeric compound according to the invention, is taken out by contacting at least one monoamine, preferably stearylamine or at least one monoisocyanate, preferably stearylisocyanate [Y] and at least one monomer [A], wherein at least a part of [A] is an amino acid or a derivative thereof, preferably selected from an ester, thioester or carboxamide, preferably of lysine, serine and/or cysteine and optionally wherein a part of [A] is a diamine, diol, or a difunctional thiol, with a diisocyanate [X], preferably selected from PDI, IPDI or MDI, and wherein the components are contacted in the presence of oil, preferably PAO or a synthetic ester and/or wherein a mechanochemical, ultrasonication and/or sonochemical method is applied, preferably ball milling, more preferably with a planetary mill.

[0071] In a preferred embodiment an excess of monomer [X] is employed in the process. More preferably the monomers [X] and [A] in the process are employed in a ratio of 1:2 to 2:1, preferably 1:1 to 2:1, more preferably 2:1.6 to 2:1.4. The ratio of [A] and [Y] preferably is 4:1 to 2:1, more preferably 3:1.

[0072] Preferably, the components are heated, more preferably until at least monomer [A] is molten, to 180 °C - 240 °C, and the other components, preferably [X], [Y] and optionally the oil are liquid. All components should be in a liquid state for reaction. Thus, the mixture can be heated to achieve melting of the amino acids or derivatives thereof. Alternatively, the amino acids or their derivatives can be dissolved in a suitable solvent and/or in the (base) oil.

[0073] Hydrophobic amino acids or their derivatives, especially hydrophobically modified amino acids may be dissolved in oil.

[0074] Up to now, the production of lubricants or lubricating greases (with the exception of solid pastes) has required high temperatures (up to approx. 180°C) and relatively long reaction times (1- 2 h).

[0075] A major problem in the determination of a suitable reaction process for in situ polymerization of amino acids in oil is posed by the high melting points of the amino acids (240 °C cysteine, 220 °C serine, 225 °C lysine). This was countered by the fact, that many biobased oils (e.g. castor oil) already have a smoke point of approx. 200 °C. If the reactions are carried out in the melt, the oil can be subjected to a considerable thermal load, which may impair the properties of the resulting oil, which can have a negative effect on the properties of the resulting lubricant. In addition, amino acids tend to decompose before their melting point is reached.

[0076] Thus, in a preferred embodiment a mechanochemical, ultrasonication and/or sonochemical method is applied in the process, preferably a mechanochemical method, wherein a mechanical force is used to achieve transformations of covalent bonds.

[0077] According to IUPAC a mechanochemical reaction is defined as a chemical reaction that is induced by the direct absorption of mechanical energy. One way to induce a mechanochemical reaction is a ball mill. These are already used for the synthesis of peptides with a low solvent content and also led to the desired results in the production of amino acid based lubricants to the desired success.

[0078] The mechanochemical method is preferably be selected from ball milling, preferably in steep mills: such as tumbling mills, short mills such as drum mills and long mills or tube mills in vibrating mills, rotor mills, mortar mills, vibrating disk mills shaker mill or a planetary mill, preferably ball milling, more preferably ball milling in a shaker mill or a planetary mill.

[0079] In preferred embodiment, a mechanochemical method is applied in the process, preferably ball milling. Such a process is preferably performed at temperatures below 50 °C, more preferably at room temperature. The reaction time can be less than 5 hours, preferably 0.5 - 3 hours, most preferably 0.2 to 1 hour. In an especially preferred embodiment, a mechanochemical method is applied in the process that is performed at temperatures below 50 °C, preferably at room temperature, for less than 5 hours, preferably 0.2 - 3 hours, most preferably 0.2 to 1 hour.

[0080] In a preferred embodiment the process comprises the steps:

- 1) providing component [Y] optionally in oil and preferably heating to the melting point of [Y], preferably at least 40 °C, more preferably 50 °C to 60 °C,

- 2) adding the mixture of step 1) to [A] and optionally steel spheres, preferably upon mixing,
- 3) adding component [X].

[0081] Optionally, in step 2) the mixture can be heated between 180 °C and 240 °C.

[0082] After addition of component [X] the mixture preferably is treated with a mechanochemical method, preferably by ball milling, more preferably with a planetary mill.

[0083] The oil preferably used in the process can be a mineral oil, natural oil such as castor oil, palm oil, coconut oil, canola oil, soybean oil or sunflower oil, or synthetic oil such as poly- α -olefin (poly- α -olefin, PAO), synthetic esters derived from the reaction of oxoacid with a hydroxyl compound such as an alcohol or phenol, or polyalkylene glycols (PAG), preferably wherein the fatty acid has at least one hydroxyl group. Preferably the oil is selected from the group of biobased oils such as natural oils, synthetic esters or PAO, more preferably synthetic esters or PAO, especially preferred synthetic esters

[0084] The raw materials used to make synthetic esters can be linear, branched, saturated, unsaturated, monofunctional, difunctional or polyfunctional. Synthetic esters for the application as lubricating agents can be purchased from a plurality of manufacturers. Synthetic esters can be selected from adipate esters, such as diisodecyl adipate (DIDA), diisononyl adipate (DINA) and dioctyl adipate (DOA), or maleate esters, such as dibutyl maleate (DBM) and dioctyl maleate (DOM) or phthalate esters, such as butyl benzyl phthalate (BBP), dibutyl phthalate (DBP), diethyl phthalate (DEP), diisobutyl phthalate, diisodecyl phthalate (DIDP), diisononyl phthalate (DINP), dioctyl phthalate (DOP), dioctyl terephthalate (DOTP) and diundecyl phthalate (DUP) or linear 911P sebacate esters such as dibutyl sebacate (DBS), dimethyl sebacate (DMS) and dioctyl sebacate (DOS) or trimellitate esters, such as triisononyl trimellitate (TINTM) and trioctyl trimellitate (TOTM).

[0085] In an alternative embodiment, biodegradable esters can be used, such as adipate, neopolyol, phthalate and trimellitate esters.

[0086] In an especially preferred embodiment at least a part of monomers [A] in the process have at least one carboxyl group or a derivative thereof, preferably a functional group selected from a carboxylate ester, thioester group or carboxamide, preferably a carboxamide, according to formula (III), wherein Z is selected from O, NH or S, preferably NH and R is hydrocarbon radical, preferably an aliphatic linear hydrocarbon radical, more preferably, wherein Z and R are selected in dependence from the oil used.

[0087] Depending on the polarity of the base oil, it is advantageous to modify the acid function by means of a side chain, which makes the thickener system more polar or less polar (hydrophobic amino acid). In addition, the side chain can be used to incorporate useful functionalities into the oligomeric or polymeric compound by means of the side chain.

[0088] Another object of the invention is a component for a lubricant, such as a thickener, or lubricant produced by a process according to the invention and a lubricant, preferably a lubricating grease comprising an oligomeric or polymeric compound according to the invention. The oligomeric or polymeric compound may act as thickener, lubricating agent and/or additive in the lubricant, preferably the lubricating grease, depending on the components, especially monomer [A].

[0089] Preferably the amount of oil in the process and/or in the lubricant is at least 80 % by weight, preferably 90 % by weight to 95 % by weight, more preferably 90 % per weight to 92 % per weight based on the total composition.

[0090] The lubricant comprising the oligomeric or polymeric compound may contain further additives, such as boundary lubricity additives that enhance fluid lubricity by adsorbing on the metal surface to form a film, limiting metal-to-metal contact. Examples include lard and canola oil. Solid lubricants can also be used for boundary lubrication; or extreme pressure additives, that are a special type of boundary lubricity additive that form a metal salt layer between mating surfaces that limit friction, wear and damage. Examples include, zinc dialkyl dithiophosphate (ZDDP), chlorinated paraffins, sulfurized lard oils, phosphate esters, overbased calcium sulfonates; or corrosion inhibitors, that prevent the fluid from corroding machine surfaces, metal work piece, cutting tool and machine tool. Examples include overbased sulfonates, alkanolamides, aminoborates, aminocarboxylates; or reserve alkalinity additives, that essentially serve as a buffer, neutralizing acidic contaminants to preserve the fluid's corrosion protection and maintain the pH in a suitable range. Examples include alkanolamines like monoethanolamine (MEA), triethanolamine (TEA), aminomethylpropanol (AMP), 2-(2-aminoethoxy) ethanol; or metal deactivators, that prevent the lubricant from staining nonferrous alloys (such as copper and brass) and reduce corrosion when dissimilar metals contact each other. They act by forming a protective coating on the metal surface. Examples include mercaptobenzothiazole, tolyltriazole, benzotriazole; or detergents, that stabilize dirt and wear debris in oil formulations; or emulsifiers reducing interfacial tension between incompatible components by forming micelles, thereby stabilizing oil-soluble additives in water-dilutable lubricants. These micelles - droplets in a colloidal system - then can remain suspended in the fluid. In lubricants, examples include sodium petroleum sulfonate and alkanolamine salts of fatty acids, or couplers, that help stabilize water-dilutable lubricants in the concentrate to prevent component separation. Couplers facilitate formation of soluble oil emulsions. Examples include propylene glycol, glycol ethers, nonionic alkoxylates; or chelating Agents (also known as water softeners or conditioners), that reduce the destabilizing effect of hard water (calcium and magnesium ions) on lubricant emulsions. An example might be ethylenediaminetetracetic acid (EDTA); or antimist additives, that minimize the amount of lubricant that disperses

into the air during machining. They are typically polymers and/or wetting agents. For oilbased systems, ethylene, propylene copolymers and polyisobutenes are used. For water-based systems, polyethylene oxides are common; or dyes, that change the color of the lubricant, usually as requested by the customer. In water-diluted fluids, their main value is to indicate that product is present, since some of these can be clear and waterlike in appearance.

[0091] In an especially preferred embodiment, the lubricant comprising the oligomeric or polymeric compound does only comprise biobased additives or preferably does not comprise any further additives.

[0092] Preferably the component for a lubricant or lubricant has an amount of decomposition product, preferably of the decomposition of monomer [A], that is below 5 % per weight, more preferably from 0 to 3 % per weight, most preferably from 0 to 1 % per weight calculated to the total composition.

[0093] Such low decomposition products can be reached by applying the process according to the invention employing a mechanochemical method and at low temperatures.

[0094] Another object of the invention is the use of an oligomeric or polymeric compound according to the invention or a component for a lubricant according to the invention as thickening agent and/or additive for lubricants.

[0095] Another object of the invention is the use of a ball mill for the production of a lubricant, preferably in a process according to the invention.

Examples

[0096] The tert-butyloxycarbonyl protecting group or tert-butoxycarbonyl protecting group (BOC group) is a protecting group.

BOC (tert-Butyloxycarbonyl) 99%	Sigma Aldrich
N,N-dimethylpyridin-4-amine	TCI
Stearylamine > 99%	DHW Ecogreen
1-octylamine	TCI
Aniline 99%	TCI
3-(Ethyliminomethylideneamino)-N,Ndimethyl-propane-1-amine hydrochloride	Sigma Aldrich
Dichloromethane >99,8%	VWR
HCl 37%	VWR
NaHCO ₃ >99,5%	VWR
NaCl > 99%	VWR
TFA 25% in water	TCI
Castor oil	Carl Roth
L-lysine >98% (TLC)	BLD Pharm
Pentamethylene N,N-diisocyanate (PDI)	Covestro

1. Process for the preparation of various derivatives of natural amino acids

[0097] The process is exemplified by the amino acids L-cysteine and the three aryl side chains C₈H₁₇, C₁₈H₃₇ and the aryl side chain C₆H₅, which can be linked to the corresponding acid function of the amino acid.

Step 1: Synthesis of the protected analogues

[0098] Boc-Lys(Boc)-OH (1 eq.), N,N-dimethylpyridin-4-amine (DMAP, 1.5 eq.) and the respective amine (stearylamine, 1-octylamine, or aniline, 1.2 eq.) were prepared under argon in dry dichloromethane (DCM, 25 mL/g Boc-Lys(Boc)-OH). After complete dissolution the mixture was cooled to 0 °C with an ice bath. 3-(Ethyliminomethylideneamino)-N,Ndimethyl-propane-1-amine hydrochloride (EDCxHCl, 1.5 eq.) was also dissolved in dry dichloromethane (DCM, 25 mL/g Boc-Lys(Boc)-OH) and added to the reaction solution through a dropping funnel and added upon stirring. The amount of the corresponding reaction components is recorded in Table 1 below:

Table 1: Listing of reaction components using the example of the synthesis of side group modified lysine.

Example	Component	M (g/mol)	Parts	n (mol)	m (mg)
1	Boc-Lys(Boc)-OH	346.42	1	2.89	2000.00
	DMAP	122.17	1.5	8.66	1057.98
	stearylamine	269.51	1.2	6.93	1867.14
	EDCxHCL	191.70	1.5	8.66	1660.1
2	Boc-Lys(Boc)-OH	346.42	1	2.89	2000.00
	1-octylamine	122.17	1.5	8.66	1057.98
	DMAP	129.24	1.2	6.93	895.36
	EDCxHCL	191.70	1.5	8.66	1660.1
3	Boc-Lys(Boc)-OH	346.42	1	2.89	2000.00
	aniline	122.17	1.5	8.66	1057.98
	DMAP	93.13	1.2	6.93	645.2
	EDCxHCL	191.70	1.5	8.66	1660.1

[0099] The reaction solution was stirred at room temperature for 24 hours. At the end of the reaction time, water (20 mL/g Boc-Lys(Boc)-OH) was added to the reaction mixture and extracted with DCM (2x 40 mL/g Boc-Lys(Boc)-OH). The organic phase was then washed with 5% HCl (40 mL/g Boc-Lys(Boc)-OH), saturated NaHCO₃ solution (40 mL/g Boc-Lys(Boc)-OH) and saturated NaCl solution (40 mL/g Boc-Lys(Boc)-OH). The organic phase was finally dried with MgSO₄. The solvent was removed by rotary evaporator and the intermediate was completely dried at high vacuum. The intermediates were obtained in the following amounts.

Table 2: Results of the intermediates from step 1 using the example of the synthesis of side group modified lysine.

Example	M g/mol
1	597.93
2	457.66
3	421.54

Step 2: Deprotection of the products from step 1

[0100] The intermediate was dissolved in a 1:2 TFA:DCM mixture (v:v, 10 mL/mmol intermediate) and stirred for 3 hours at room temperature. Subsequently solvent was removed with a rotary evaporator and the sample was dried overnight under high vacuum.

[0101] The residue was mixed with DCM (10 mL/mmol intermediate) and extracted with 1M NaOH (2x 10 mL/mmol intermediate). The aqueous phase was extracted again with DCM (10 mL/mmol intermediate). Subsequently, the organic phases were combined and dried with MgSO₄ and filtered. The solvent was removed by rotary evaporator and the product was dried at high vacuum. The products were obtained as follows:

Table 3: Results of products of synthesis of side group modified lysine

Example	M (g/mol)	Purity (%)
1	397.69	95.6
2	257.42	95.7
3	221.30	92.3

2. Process for the preparation of a component for a lubricant or a lubricant - In situ synthesis using a ball mill

Example 4:

[0102] In a 25 mL beaker, 5 mL of castor oil and stearylamine were heated to the melting point (setting 120 °C). In a

15 mL mixing vessel with stirring unit, L-lysine (Lys) was introduced and 9 steel spheres (diameter 6 mm) were added. Subsequently, the mixture of castor oil and stearylamine was added and stirred for four minutes. Then, pentamethylene N,N-diisocyanate (PDI) was added and stirred for a further ten minutes stirred. The resulting lubricant was cooled to room temperature and then rolled.

[0103] To homogenize the lubricant, it was rolled using the EXAKT 35 three-roll mill from EXAKT Apparatebau. The roll diameter was 35 mm, the roll width 100 mm. The working width could be adjusted between 30 and 80 mm could be adjusted. In this case, the smallest working width of 30 mm was used. The respective amounts of the reaction components are listed in the following table.

Table 4: Exemplary weights of the reaction components

Example	Component	M (g/mol)	Parts	n (mmol)
5	PDI	154.17	1	1.43
	L-Lysine	146.19	0.75	1.07
	stearylamine	269.52	0.25	0.36
	castor oil (V = 5000 μ L)			
6	POI	154.17	1	0.91
	LysNHC18	146.19	0.75	0.68
	stearylamine	269.52	0.25	0.23
	castor oil (V = 5000 μ L)			
7	POI	154.17	1	1.14
	LysNHC8	146.19	0.75	0.86
	stearylamine	269.52	0.25	0.29
	castor oil (V = 5000 μ L)			
8	POI	154.17	1	1.12
	LysNPh	146.19	0.75	0.92
	stearylamine	269.52	0.25	0.31
	castor oil (V = 5000 μ L)			

Example 5a:

[0104] As example 5, except that all reaction components were added to the mixing vessel at room temperature.

Example 5b:

[0105] Same as Example 5a except that a planetary mill with a capacity of 250 ml and 125 g spheres was used.

Examples 6-8:

[0106] Same as Example 5b, only with chemically modified amino acids. Above examples were also successful employing cysteine and serine and their respective derivatives as amino acid compound in [A].

Claims

1. Oligomeric or polymeric compound, preferably for application in lubricants, consisting of

- at least one monomeric unit X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and
- two units Y, derived from an organic compound [Y] that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group, that is no carboxyl group or a derivative thereof, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group, and optionally
- at least one monomeric unit A, derived from a monomer [A] featuring two functional groups, that react per

addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, wherein at least a part of the monomer [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxamide group, and wherein [Y] has a primary amine group and at least one carboxamide group, if the oligomeric or polymeric compound does not contain A.

2. Oligomeric or polymeric compound, according to claim 1, consisting of

- at least two monomeric units X, derived from an isocyanate monomer [X] with at least two isocyanate groups, preferably a diisocyanate to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, and
- two units Y, derived from an organic compound [Y] that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a secondary amine group, and
- at least one monomeric unit A, derived from a monomer [A] that has two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the two functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomers [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxamide group.

3. Oligomeric or polymeric compound according to claim 2, **characterized in that** the two functional groups that react per addition reaction with isocyanate groups, that are no carboxyl groups or derivatives thereof, of monomer [A] are not bound to the carboxamide group.

4. Oligomeric or polymeric compound, according to claims 1 to 3, **characterized in that** X is a monomeric unit according to the formula $-\text{[OCNH-R}_2\text{-NHCO]}-$, wherein R_2 is a divalent hydrocarbon radical.

5. Oligomeric or polymeric compound, according to claims 1 to 4, **characterized in that** the two units Y, are organic moieties according to the formula $[\text{R}_3\text{-Z}_3]-$, wherein $\text{Z}_3 = \text{O, S, N, NH or NHCO}$ and wherein R_3 is a monovalent organic moiety that optionally has a carboxamide group, preferably a monovalent hydrocarbon radical, preferably derived from monofunctional amines, alcohols, thiols or isocyanates or hydrophobic amino acids [Y].

6. Oligomeric or polymeric compound, according to claims 2 to 5, **characterized in that** A is a monomeric unit according to the formula $-\text{[Z}_1\text{-R}_1\text{-Z}_1]-$ wherein Z_1 is the same or different and is selected from O, S, N or NH, and wherein R_1 is an organic moiety that optionally has a carboxamide group, preferably a monovalent hydrocarbon radical or a carboxamide.

7. Oligomeric or polymeric compound according to claims 2 to 6, with Y according to the formula $[\text{R}_3\text{-Z}_3]-$, wherein R_3 is a monovalent hydrocarbon radical,

according to formula (I), when $\text{Z}_3 = \text{O, S, N, NH}$
or according to formula (II), when $\text{Z}_3 = \text{NHCO}$,



8. Oligomeric or polymeric compound according to claim 7, according to formula (I).

9. Oligomeric or polymeric compound according to claim 7 or 8, **characterized in that** n is an integer from 1 to 20, preferably from 2 to 15, more preferably from 5 to 10, especially preferred 9.

10. Oligomeric or polymeric compound according to any one of claims 1 to 9, **characterized in that** at least a part of monomers [A], preferably monomers [A], are amino acid amides, more preferably amino acid amides deriving from

natural amino acids, and optionally a part of monomers [A] is selected from diamines, bifunctional thioles or dioles, preferably aliphatic diamines, bifunctional thioles or dioles.

11. Process for production of a component of a lubricant, preferably of a lubricant, more preferably comprising an oligomeric or polymeric compound according to any of the claims 1 to 10, by contacting component [Y], wherein

[Y] is an organic compound that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group, that is no carboxyl group or a derivative thereof, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group,

and optionally monomer [A], wherein [A] has two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxyl group or a derivative thereof, preferably a functional group selected from a carboxyl group, carboxylate ester, thioester group or carboxamide, and wherein

[Y] has a primary amine group and additionally at least carboxyl group or a derivative thereof, preferably a functional group selected from a carboxyl group, carboxylate ester, thioester group or carboxamide, if no monomer [A] is added, with component [X], wherein [X] is at least one isocyanate monomer with at least two isocyanate groups, preferably a diisocyanate monomer according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent, hydrocarbon radical, wherein the components are contacted in the presence of oil and/or wherein a mechanochemical, ultrasonication and/or sonochemical method is applied.

12. Process for production of a component of a lubricant, preferably of a lubricant, more preferably comprising an oligomeric or polymeric compound according to any of the claims 2 to 10, by contacting components [Y] and [A], wherein

[Y] is an organic compound that has one isocyanate group or one functional group that reacts per addition reaction with an isocyanate group, that is no carboxyl group or a derivative thereof, preferably selected from a primary or secondary amine group, a sulfanyl group or hydroxyl group, especially preferred a primary or secondary amine group, and wherein

monomer [A] has two functional groups, that react per addition reaction with isocyanate groups and that are no carboxyl groups or derivatives thereof, wherein the functional groups are the same or different and preferably selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups, and wherein at least a part of the monomer [A] has a first functional group selected from primary or secondary amine groups, sulfanyl groups or hydroxyl groups and a second functional group that is a primary amine group and additionally at least one carboxyl group or a derivative thereof, preferably a functional group selected from a carboxyl group, carboxylate ester, thioester group or carboxamide, with

component [X], wherein [X] is at least one isocyanate monomer with at least two isocyanate groups, preferably a diisocyanate monomer according to the formula $\text{OCN-R}_2\text{-NCO}$, wherein R_2 is a divalent hydrocarbon radical, wherein the components are contacted in the presence of oil and/or wherein a mechanochemical, ultrasonication and/or sonochemical method is applied.

13. Process for production of a component of a lubricant according to claims 11 or 12, **characterized in that** the mixture of the components and preferably the oil is heated to a temperature of 180 °C to 240 °C.

14. Process for production of a component of a lubricant, preferably of a lubricant, preferably according to claims 11 or 12, **characterized in that** a mechanochemical, ultrasonication and/or sonochemical method is applied, preferably a mechanochemical method, wherein a mechanical force is used to achieve transformations of covalent bonds and wherein the components are contacted in the presence of oil or a solvent, preferably oil.

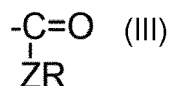
15. Process for production of a component of a lubricant according to claim 14, **characterized in that** the mechanochemical method is selected from ball milling, preferably in steep mills: such as tumbling mills, short mills such as drum mills and long mills or tube mills in vibrating mills, rotor mills, mortar mills, vibrating disk mills shaker mill or a planetary mill, preferably ball milling, more preferably ball milling in a shaker mill or a planetary mill.

16. Process for production of a component of a lubricant, preferably according to claim 14 or 15, **characterized in that**

it is performed at temperatures below 50 °C, preferably at room temperature, more preferably at room temperature for less than 5 hours, preferably 0.2 - 3 hours, most preferably 0.2 to 1 hour.

17. Process for production of a component of a lubricant according to claims 11 to 13 or 14 to 16, **characterized in that** the oil is a synthetic oil such as poly-alpha-olefin (poly- α -olefin, PAO), synthetic esters derived from the reaction of oxoacid with a hydroxyl compound such as an alcohol or phenol.

18. Process for production of a component for lubricant according to claims 11 to 13 or 14 to 17, **characterized in that** at least a part of monomers [A] have at least one carboxyl group or a derivative thereof, preferably a functional group selected from a carboxyl group, carboxylate ester, thioester group or carboxamide, preferably a carboxamide, according to formula (III):



wherein Z is selected from O, NH or S, and R is an aliphatic hydrocarbon radical, more preferably, wherein Z and R are selected in dependence from the oil used.

19. Component for a lubricant or lubricant produced by a process according to any one of claims 11 to 13 or 14 to 18.

20. Lubricant comprising an oligomeric or polymeric compound according to claims 1 to 10 or a component for lubricant or a lubricant produced by a process according to any one of the claims 11 to 13 or 14 to 18.

21. Component for a lubricant or lubricant according to claim 19 or 20, **characterized in that** it has an amount of decomposition product, preferably of the decomposition of monomer [A], that is below 5 % per weight, more preferably from 0 to 3 % per weight, most preferably from 0 to 1 % per weight calculated to the total composition.

22. Use of an oligomeric or polymeric compound according to claims 1 to 10 or a component for a lubricant according to claim 19 as thickening agent and/or additive for lubricants.

23. Use of a ball mill for the production of a lubricant, preferably in a process according to claims 11 to 18.



PARTIAL EUROPEAN SEARCH REPORT

Application Number

under Rule 62a and/or 63 of the European Patent Convention.
This report shall be considered, for the purposes of
subsequent proceedings, as the European search report

EP 22 21 4519

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
A,P	March Jerry ET AL: "Carboxylic acid - Nitriles, Synthesis, Reactions ; Britannica", Encyclopedia Britannica, 23 August 2023 (2023-08-23), XP093075721, Retrieved from the Internet: URL:https://www.britannica.com/science/carboxylic-acid/Nitriles [retrieved on 2023-08-23] * the whole document * ----- -/--	2-10, 12-23	INV. C10M115/08 C10M119/24 C10M121/04 C10M159/12 C07C275/00 ADD. C10N30/00 C10N50/10 C10N70/00
			TECHNICAL FIELDS SEARCHED (IPC)
			C10M C10N
INCOMPLETE SEARCH			
The Search Division considers that the present application, or one or more of its claims, does/do not comply with the EPC so that only a partial search (R.62a, 63) has been carried out.			
Claims searched completely :			
Claims searched incompletely :			
Claims not searched :			
Reason for the limitation of the search: see sheet C			
Place of search		Date of completion of the search	Examiner
Munich		24 August 2023	Grefß, Tobias
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			

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DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (IPC)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
A	ZHELEZNYI LEONID ET AL: "Development of compositions of urea greases on aminoamides of fatty acids", EASTERN-EUROPEAN JOURNAL OF ENTERPRISE TECHNOLOGIES, [Online] vol. 3, no. 6 (87), 1 January 2017 (2017-01-01), pages 9-14, XP093075999, ISSN: 1729-3774, DOI: 10.15587/1729-4061.2017.99580 Retrieved from the Internet: URL:https://pdfs.semanticscholar.org/56cb/dec67e0e9f5b99a64f10001bc1c2b7db7149.pdf> [retrieved on 2023-08-23] * p. 10-11, item 4; fig. 5; p. 13, item 7 *	2-10, 12-23	
A	----- US 3 769 212 A (STANTON G ET AL) 30 October 1973 (1973-10-30) * claims 9-11 * -----	2-10, 12-23	TECHNICAL FIELDS SEARCHED (IPC)



**INCOMPLETE SEARCH
SHEET C**

Application Number

EP 22 21 4519

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Claim(s) searched incompletely:

2-10, 12-23

Claim(s) not searched:

1, 11

Reason for the limitation of the search:

Rule 62a(1) and 63(1) EPC

ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.

EP 22 21 4519

5 This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

24-08-2023

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15	US 3769212	A	30-10-1973	NONE
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82