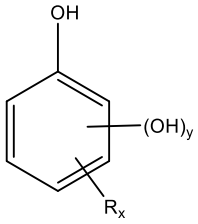
CLAIMS

1. A process for the preparation of prills of compounds of formula (A)



Formula (A)

wherein:

- y = 0 or 1,
- R represents a substituent selected from the group consisting of an alkyl group, an alkenyl group, an alkoxy group, a hydroxyalkyl group, an alkoxyalkyl group, a cycloalkyl group, an aryl group, an aryloxy group, an arylalkyl group, a hydroxyl group, a nitro group, a halogen atom, a halo- or perhaloalkyl group, a formyl group, an acyl group, a carboxylic group, an amino group and an amido group
- x = 0, 1, 2, 3 or 4

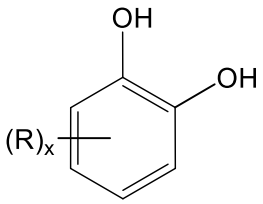
with the proviso that if y = 0 then x = 1 and R is an alkoxy group in para position to the hydroxyl group, said process comprising:

- providing a molten composition comprising from 50 to 100 wt.%, preferably from 75 to 100 wt.% and more preferably from 90 to 100 wt.%, of a compound of formula (A) and less than 0.1 wt.% of water;
- forcing said molten composition through at least one droplet generator means to form droplets;
- cooling said droplets to form solid diphenol derivative prills,

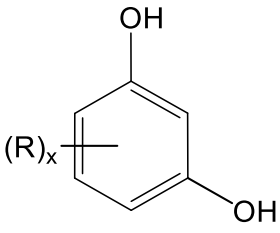
wherein the cooling is carried out by:

- a cooling medium which is a cooling gas or a liquefied inert gas, or
- two cooling media wherein the first cooling medium is a liquefied inert gas and the second cooling medium is a cooling gas or the first and the second cooling medium are a cooling gas.

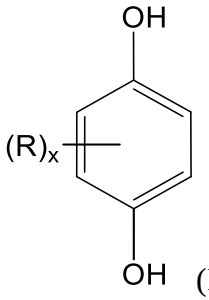
2. The process according to Claim 1, wherein the diphenol compound(s) is (are) a compound according to the general formula (I), (II) or (III)



(I)



(II)



(III)

wherein x represents 0, 1, 2, 3 or 4, and each R independently represents a substituent selected from the group consisting of an alkyl group, an alkenyl group, an alkoxy group, a hydroxyalkyl group, an alkoxyalkyl group, a cycloalkyl group, an aryl group, an aryloxy group, an arylalkyl group, a hydroxyl group, a nitro group, a halogen atom, a halogeno- or perhalogenoalkyl group, a formyl group, an acyl group, a carboxylic group, an amino group and an amido group.

3. The process according to Claim 1 to 2, wherein the diphenol compound(s) is (are) selected from the group consisting of pyrocatechol (PC), hydroquinone (HQ), resorcinol (Res), pyrogallol (Py), 4-tert-butyl-catechol (TBC) and tert-butyl-hydroquinone (TBHQ), more preferably the diphenol compound is hydroquinone or a mixture of hydroquinone with at least one other diphenol compound.

4. The process according to any one of Claims 1 to 3, wherein said molten composition comprises from 50 to 100 wt.% of a diphenol compound or a mixture of at least two diphenol compounds and from 0 to 50 wt.% of one or several other compounds, wherein said other compounds are selected from the group consisting of:

- diphenol derivatives, especially diphenol ether, for instance para-methoxyphenol (PMP), para-phenoxy-phenol (PPP), dibutylhydroxyanisole (BHA), 2,6-ditertbutyl-4-methoxyphenol (DTBHA), vanillin, ethyl vanillin, vanillin alcohol, vanillic acid, and paradimethoxybenzene (PDMB);

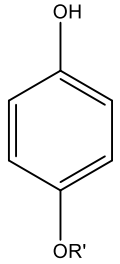
- phenol derivatives, especially alkylated phenols, for instance dibutylhydroxytoluene (BHT) and 2,4-dimethyl,6-tertbutyl-phenol (TOPANOL A);
- other organic compounds, especially cyclic or heterocyclic compounds, for instance phenothiazine (PTZ), derivatives of TEMPO, preferably tetramethyl piperidinyloxy (TEMPO), hydroxyl-tetramethyl piperidinyloxy (TEMPO-OH), 4-oxo-2,2,6,6-tetramethyl-1-piperidinyloxyl (4-Oxo-TEMPO), benzoquinone (PBQ), benzoic acid.

5. The process according to Claim 4, wherein said molten composition comprises:

- from 50 to 100 wt.%, preferably from 75 to 100 wt.% and more preferably from 90 to 100 wt.% of hydroquinone, and
- from 0 to 50 wt.%, preferably from 0 to 25 wt.%, and more preferably from 0 to 10 wt.%, of para-methoxyphenol, resorcinol, para-phenoxy-phenol, pyrocatechol, dibutylhydroxytoluene, tert-butyl-hydroquinone, 2,6-ditertbutyl-4-methoxyphenol and phenothiazine or mixtures thereof.

6. The process according to claim to any one of claims 1 to 2 wherein said molten composition comprises from 50 to 100 wt.% of a diphenol compound or a mixture of at least two diphenol compounds and from 0 to 50 wt.% of one or several other compounds, wherein said other compounds are selected from the group consisting of tocopherol derivatives, and complexes of copper having an oxidation state of 2, preferably copper dibutyl dithiocarbamate or copper acetate, 2-sec-butyl-4,6-dinitrophenol or p-phenylenediamine.

7. A process for the preparation of prills of diphenol derivative of formula (IV)



Formula (IV)

wherein R’ represents an alkyl group or an aryl group, comprising:

- providing a molten composition comprising from 50 to 100 wt.%, preferably from 75 to 100 wt.% and more preferably from 90 to 100 wt.%, of the diphenol derivative compound, and less than 0.1 wt.% of water;
- forcing said molten composition through at least one droplet generator means to form droplets;
- cooling said droplets to form solid prills of the diphenol derivative.

8. The process according to claim 7 wherein the diphenol derivative according to formula (IV) is selected from the group consisting of para-methoxyphenol (PMP, para-ethoxyphenol (PEP) or para-phenoxyphenol (PPP).

9. The process according to any one of Claims 7 to 8, wherein said molten composition comprises from 50 to 100 wt.% of a diphenol derivative according to fomula (IV) and from 0 to 50 wt.% of one or several other compounds, wherein said other compounds are selected from the group consisting of:

- diphenol derivatives, especially diphenol ether, for instance, para-phenoxy-phenol (PPP), dibutylhydroxyanisole (BHA), 2,6-ditertbutyl-4-methoxyphenol (DTBHA), vanillin, ethyl vanillin, vanillin alcohol, vanillic acid, and paradimethoxybenzene (PDMB);
- phenol derivatives, especially alkylated phenols, for instance dibutylhydroxytoluene (BHT) and 2,4-dimethyl,6-tertbutyl-phenol (TOPANOL A);
- other organic compounds, especially cyclic or heterocyclic compounds, for instance phenothiazine (PTZ), derivatives of TEMPO, preferably tetramethyl piperidinyloxy (TEMPO), hydroxyl-tetramethyl piperidinyloxy (TEMPO-OH), 4-oxo-2,2,6,6-tetramethyl-1-piperidinyloxyl (4-Oxo-TEMPO), benzoquinone (PBQ), benzoic acid.

10. The process according to Claim 9, wherein said molten composition comprises:

- from 50 to 100 wt.%, preferably from 75 to 100 wt.% and more preferably from 90 to 100 wt.% of para-methoxyphenol, and
- from 0 to 50 wt.%, preferably from 0 to 25 wt.%, and more preferably from 0 to 10 wt.%, of phenothiazine, α-tocopherol, 2-sec-butyl-4,6-dinitrophenol, p-phenylenediamine or mixtures thereof.

11. The process according to any one of Claims 1 to 10, wherein said molten composition further comprises at least one additive selected from the group consisting of antioxidants, antifoams, rust inhibitors, corrosion inhibitors, surfactants, detergents, dispersants, antifouling additives, and anti-deposition agents.

12. The process according to any one of Claims 1 to 11, wherein the cooling is carried out by a cooling medium which is a liquefied inert gas, preferably liquid nitrogen, and said cooling medium flows co-currently with respect to the droplets of the diphenol composition.

13. The process according to any one of Claims 1 to 11, wherein the cooling is carried out by a cooling medium which is a cooling gas, preferably an inert gas, more preferably depleted air or nitrogen, at a temperature of between -40°C and +100°C, preferably between -20°C and 20°C, and said cooling medium flows counter-currently with respect to the droplets of the diphenol composition.

14. The process according to any one of Claims 1 to 13, wherein the cooling is carried out by two cooling media: the droplets are first cooled by a liquefied inert gas, preferably liquid nitrogen and secondly by a cooling gas.

15. The process according to any one of claims 1 to 14 further comprising a step comprising separating fine particles of diphenol prills having a size of less than 100 µm, preferably by sieving or cycloning, and recycling said fine particles.

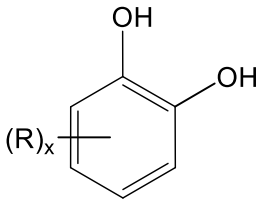
16. Diphenol prills selected from the group consisting of:

- diphenol prill comprising from 50 to 100 wt.% of a diphenol compound or a mixture of at least two diphenol compounds, having a spherical shape and a mean particle diameter d_{50} of between 0.3 mm and 1 cm, and having a water content of strictly less than 1000 ppm;

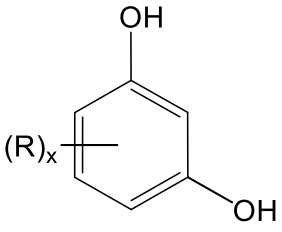
- diphenol prills comprising from 50 to 100 wt.% of a mixture of at least two diphenol compounds, having a spherical shape and a mean particle diameter d_{50} of between 0.3 mm and 1 cm; and

- diphenol prills comprising from 50 to 99.9 wt.% of a diphenol compound or a mixture of at least two diphenol compounds, and from 0.1 to 50 wt.% of one or several other compounds, having a spherical shape and a mean particle diameter d_{50} of between 0.3 mm and 1 cm, and having a water content less than 1000 ppm,

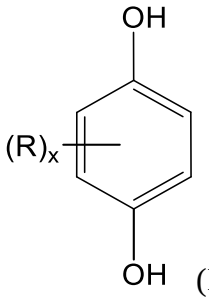
wherein the diphenol compounds are represented by formula (I), (II) or (III)



(I)



(II)



(III)

wherein x represents 0, 1, 2, 3 or 4, and each R independently represents a substituent selected from the group consisting of an alkyl group, an alkenyl group, an alkoxy group, a hydroxyalkyl group, an alkoxyalkyl group, a cycloalkyl group, an aryl group, an aryloxy group, an arylalkyl group, a hydroxyl group, a nitro group, a halogen atom, a halogeno- or perhalogenoalkyl group, a formyl group, an acyl group, a carboxylic group, an amino group and an amido group.

17. Prills of diphenol derivative according to formula (IV), comprising at least 98 wt%, more preferably at least 99 wt% of a diphenol derivative according to formula (IV), preferably para-methoxyphenol.

18. Prills of diphenol derivative according to formula (IV), comprising:

- from 50 to 100 wt.%, preferably from 75 to 100 wt.% and more preferably from 90 to 100 wt.% of para-methoxyphenol, and

- from 0 to 50 wt.%, preferably from 0 to 25 wt.%, and more preferably from 0 to 10 wt.%, of phenothiazine, α-tocopherol, 2-sec-butyl-4,6-dinitrophenol, p-phenylenediamine or mixtures thereof.

19. Diphenol prills according to any one of Claims 16 to 18, wherein said mean particle diameter d_{50} is of between 0.4 mm and 5 mm, preferably of between 0.5 mm and 3 mm, and more preferably between 0.8 mm and 2 mm.

20. Diphenol prills according to any one of Claims 16 to 19, having a hardness of at least 1 N.

21. Diphenol prills according to any one of Claims 16 to 20, having a friability of less than 15%.

22. Diphenol prills according any one of Claims 16 to 21, comprising:

- from 50 to 100 wt.%, preferably from 75 to 100 wt.% and more preferably from 90 to 100 wt.% of hydroquinone, and
- from 0 to 50 wt.%, preferably from 0 to 25 wt.%, and more preferably from 0 to 10 wt.%, of para-methoxyphenol, resorcinol, para-phenoxy-phenol, pyrocatechol, dibutylhydroxytoluene, tert-butyl-hydroquinone, 2,6-ditertbutyl-4-methoxyphenol and phenothiazine or mixtures thereof.

23. Diphenol prills according any one of Claims 16 to 22, comprising:

- from 50 to 100 wt.% of a diphenol compound or a mixture of at least two diphenol compounds, and
- from 0 to 50 wt.% of one or several other compounds, wherein said other compounds are selected from the group consisting of tocopherol derivatives, and complexes of copper having an oxidation state of 2, preferably copper dibutyl dithiocarbamate or copper acetate, 2-sec-butyl-4,6-dinitrophenol or p-phenylenediamine.

24. Diphenol prills according to any one of Claims 16 to 23, further comprising at least one additive selected from the group consisting of antioxidants, antifoams, rust inhibitors, corrosion inhibitors, surfactants, detergents, dispersants, antifouling additives, and anti-deposition agents.

25. Use of the diphenol prills according to any one of Claims 16 to 24 as antioxidant, polymerization inhibitor or as building block for the synthesis of organic or inorganic compound, for instance as a monomer for polymer production.