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(54) Title: PROCESS FOR EXTRACTION OF CURCUMINOIDS

(57) Abstract: A process (100/200/300/400) for extraction of curcuminoids is disclosed. The process (100/200/300/400) extracts curcuminoids finely powdered turmeric as a raw material using solvents such as supercritical fluid and acetic acid with a time tested human use that leaves no trace in the extract after conversion to the gaseous state. The process (100/200/300/400) results in extraction of solvent and heavy metal free nutraceuticals such as curcuminoids which may also be in a nanoparticle form.



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PROCESS FOR EXTRACTION OF CURCUMINOIDS

FIELD OF THE INVENTION

5 The present invention generally relates to a process for extraction of bioactives and nutraceuticals and more particularly to a process for extraction of solvent and heavy metal free bioactives and nutraceuticals such as curcuminoids in a powder form which can be in micro or nano size range.

10 BACKGROUND OF THE INVENTION

Nutraceuticals such as curcuminoids or simply curcumin that comprise of curcumin I, II, and III are extracted industrially using a two stage process that involves following general steps: i) Treating powdered turmeric with a non polar solvent such as hexane
15 followed by treatment of the turmeric powder with a medium polar solvent such as ethyl acetate; or ii) Treating powdered turmeric with a medium polar solvent such as ethyl acetate followed by treatment of the extract with another medium polar solvent such as isopropyl alcohol.

20 Current state of the art uses solvents that may pose significant health risks over longer run. Although solvents such as ethyl acetate are considered as safe, exposure to humans is relatively new and their full drawbacks are yet to emerge. Curcumin has one of the highest consumption among populations across demographic distribution for its medicinal and nutraceutical values. Hence, a process for extraction of
25 curcumin is needed that uses green solvents, specifically those with a time tested human use.

Hence, there exists a need to provide a process for extraction of solvent and heavy metal free curcuminoids that overcomes the above mentioned drawbacks of the prior art.

5 OBJECT OF THE INVENTION

An object of the present invention is to extract curcuminoids using green solvents, specifically those with a time tested human use.

10 SUMMARY OF THE INVENTION

Accordingly, the present invention provides a process for extraction of curcuminoids. As per a first embodiment, the process comprises treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with
15 co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

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The process further comprises pumping of co-solvent over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction at a temperature ranging between 32 to 150 °C, pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove non curcuminoids from the
25 de-oiled turmeric while selectively keeping curcuminoids therein. The co-solvent is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents wherein water concentration can be up to 50%, the co-solvent is preferably

acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.

5 The process further comprises extracting remaining residue from the product of above step in solvent preferably acetic acid to obtain an extract containing curcuminoids, wherein acetic acid is used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours. Other solvents that can be used include any one of hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water
10 with the above mentioned solvents wherein water concentration can be up to 50%. The process involves repeating the previous step to combine, filter and concentrate resulting extracts.

Further, the process comprises crystallizing the curcuminoids from the extract at a
15 temperature ranging between 18 -3 0 °C. Thereafter, the process comprises filtering the crystallized curcuminoids, washing for example with cold acetic acid at a temperature ranging between 18 - 30 °C and drying to obtain totally solvent and heavy metal free dried curcuminoids.

20 In a second embodiment, the process for extraction of curcuminoids comprises treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled
25 turmeric. The supercritical fluid is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

The process further comprises pumping of co-solvent over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction at a temperature ranging between 32 to 150 °C, pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove non curcuminoids from the de-oiled turmeric while selectively keeping curcuminoids therein. The co-solvent is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%, the co-solvent is preferably acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.

The process further comprises extracting remaining residue from the product of above step in solvent preferably acetic acid to obtain an extract containing curcuminoids, wherein acetic acid is used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours. Other solvents that can be used include any one of hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%.

The process further comprises adding extract obtained from the previous step in any one of cold water and hot water as a continuous flowing stream to precipitate curcuminoids and to remove water soluble impurities therefrom at a temperature ranging from 0 to 100 °C.

In a next step, the process comprises separating the precipitated curcuminoids and washing repeatedly with any one of cold water and hot water as a continuous flowing stream to obtain partially purified curcuminoids; drying the partially purified curcuminoids and thereafter putting in an extractor; pumping co-solvent with

supercritical fluid over the dried curcuminoids in three steps to precipitate almost pure curcuminoids in the extractor; and finally washing the pure curcuminoids for example with cold acetic acid to finally obtain totally solvent and heavy metal free dried curcuminoids.

5

In a third embodiment, the process for extraction of curcuminoids comprises treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg
10 of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

15 The process further comprises extracting the de-oiled turmeric with solvent preferably acetic acid, wherein acetic acid is used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours. Other solvent that can be used alternatively include any one of hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a
20 combination of water with the above mentioned solvents wherein water concentration can be up to 50%.

In a next step, the process comprises adding extract obtained from above step to water to precipitate the curcuminoids along with impurities such as non
25 curcuminoids.

The process in subsequent steps comprises filtering the precipitated curcuminoids and impurities and washing repeatedly with any one of cold water and hot water as a

continuous flowing stream to obtain partially purified curcuminoids; drying the partially purified curcuminoids and thereafter putting in an extractor; pumping co-solvent with supercritical fluid over the dried curcuminoids in three steps to precipitate almost pure curcuminoids in the extractor The co-solvent is selected from
5 any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol, or a combination of water with the above mentioned solvents wherein water concentration can be up to 50%, the co-solvent is preferably acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes. In the last step, the process involves washing the
10 almost pure curcuminoids for example with cold acetic acid at a temperature ranging between 18-30 °C to finally obtain totally solvent free dried curcuminoids.

As per a fourth embodiment, the process comprises treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with
15 co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

20

The process further comprises pumping of co-solvent over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction at a temperature ranging between 32 to 150 °C, pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove non curcuminoids from the
25 de-oiled turmeric while selectively keeping curcuminoids therein. The co-solvent is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents wherein water concentration can be up to 50%, the co-solvent is preferably

acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.

5 The process in a next step comprises extracting remaining residue from the product of above step in in solvent to obtain an extract containing curcuminoids, wherein the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours such that the resulting extract having solvent preferably acetic acid ranging from 5 to 99.5 % of the total mass. The process involves repeating the previous step
10 to combine, filter and concentrate resulting extracts having acetic acid ranging from 5 to 95.5 % of the total mass. Other solvents that can be alternatively used include any one of hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%.

15 The process in a further step comprises re-extracting the extract obtained from previous step with supercritical fluid in an extractor at temperatures between 35 to 150 °C, pressure ranging from 80-700 bars, at a flow rate ranging from 10 to 250 kg/kg of turmeric to remove non curcuminoids and acetic acid and precipitating
20 almost pure curcuminoids.

The process in subsequent steps involves washing the precipitated almost pure curcuminoids for example with cold acetic acid at a temperature ranging between 18-30 °C to obtain almost pure curcuminoids; and finally drying the almost pure
25 curcuminoids to obtain a totally solvent and heavy metal free dried curcuminoids.

DETAILED DESCRIPTION OF THE INVENTION

The foregoing objects of the invention are accomplished and the problems and shortcomings associated with the prior art techniques and approaches are overcome by the present invention as described below in the preferred embodiment.

- 5 The present invention provides a process for extraction of nutraceuticals. The process of the present invention extracts solvent and heavy metal free nutraceuticals such as curcuminoids using green solvents, specifically those with a time tested human use.

Specifically, a process for extraction of curcuminoids is described in accordance with
10 the present invention. More specifically, the process is described for extraction of solvent and heavy metal free nutraceuticals such as curcuminoids/ curcumin. However, it is understood that the process can be used for extraction of other nutraceuticals known in the art.

- 15 In a first embodiment, the present invention provides a process (100) for extraction of curcuminoids and specifically, the process (100) for extraction of solvent and heavy metal free curcuminoids from finely powdered turmeric as a raw material which is described in detailed step-wise manner herein below:

20 The process (100) begins at step (10). At step (20), the process (100) involves supercritical fluid extraction which involves treating the finely powdered turmeric (herein after 'the turmeric') with supercritical fluid to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from but not limiting to supercritical CO₂, propane, butane, R22, R134, and N₂O alone or in various
25 combinations thereof. Specifically, the turmeric is treated with supercritical CO₂ at a temperature ranging between 35 to 150 °C, preferably between 37-70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100-500 bars, more preferably between 130 to 180 bars, and at a flow

rate ranging from 10 to 250 kg/kg of the turmeric. Optionally, step (20) can also be accomplished by using co solvents such as hexane, ether, ethyl acetate, acetone, acetic acid, methanol, ethanol, isopropyl alcohol and the like in combination with supercritical fluids as per various alternate embodiments of the process (100).

5

At step (30), the process (100) involves pumping the co-solvent in a periodic/segmented manner over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction to remove non curcuminoids including but not limited to resinous material and other medium polar impurities from the de-oiled turmeric while selectively keeping curcuminoids therein. Thus, after the first step of co-solvent pumping, co-solvent pumping is repeated 2 more times after an interval ranging from 5 to 30 minutes, preferably 15 minutes till 1 hour. Specifically, the co-solvent is acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 min, preferably, 1 min at a temperature ranging between 32 to 150 °C, preferably between 37 to 70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100 to 150 bars, more preferably between 130 to 180 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric. However, it is understood that the co-solvent can also be selected from any of the common organic solvents such as acetone, ethyl acetate, methanol, ethanol, isopropyl alcohol and the like or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% as per various alternate embodiments of the process (100). Similarly, it is understood that the co solvent can be pumped in a continuous manner in which case the percentage of co-solvent may range from 1 to 40 % of the turmeric quantum and for a period ranging from 2 minutes to 2 hours depending upon the quantum of curcuminoids present in the feed as well as on the flow rate of supercritical fluid.

25

At step (40), the process (100) involves solvent extraction which involves extracting the remaining residue from the product of step (30) in a solvent to obtain an extract containing curcuminoids for further processing. In an embodiment, solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, preferably 5
5 times at temperature ranging from 35 to 80 °C, preferably 60 °C under stirring for a duration of 15 minutes to 2 hours, preferably 30 minutes or for a duration of 30 minutes to 2 hours, preferably 1 hour. However, other solvents such as hexane, ether, ethyl acetate, acetone, methanol, ethanol, and isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to
10 50% can also be used in alternate embodiments of the process (100).

At step (50), the process (100) involves optionally repeating the step (40) 2-3 times, thereafter combining the extracts, filtering and concentrating the extracts under vacuum at lower temperatures, preferably 40 °C.
15

At step (60), the process (100) involves crystallizing the curcuminoids from the concentrated extract at a temperature ranging between 18-30 °C, preferably 20 °C.

At step 70), the process (100) involves filtering the crystallized curcuminoids under a
20 filter press followed by washing for example with cold acetic acid at a temperature ranging between 18-30 °C, preferably 20 °C and thereafter drying using conventional methods of drying to obtain totally solvent and heavy metal free dried curcuminoids. In an embodiment, the totally solvent and heavy metal free curcuminoids are obtained in nanoparticle forms with 95 % purity and having following composition.
25 The process (100) ends at step (80).

In a second embodiment, the present invention provides the process (200) for extraction of curcuminoids and specifically, the process (200) for extraction of

solvent and heavy metal free curcuminoids from finely powdered turmeric as a raw material which is described in detailed step-wise manner herein below:

5 The process (200) begins at step (110). At step (120), the process (200) involves supercritical fluid extraction which involves treating the finely powdered turmeric (herein after 'the turmeric') with supercritical fluid to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from but not limiting to supercritical CO₂, propane, butane, R22, R134, and N₂O alone or in various combinations thereof. Specifically, the turmeric is treated with supercritical CO₂ at a
10 temperature ranging between 35 to 150 °C, preferably between 37-70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100-500 bars, more preferably between 130 to 180 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric. Optionally, step (120) can also be accomplished by using co solvents such as hexane, ether, ethyl acetate, acetone,
15 acetic acid, methanol, ethanol, isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%, and the like in combination with supercritical fluids as per various alternate embodiments of the process (200).

20 At step (130), the process (200) involves pumping the co-solvent in a periodic/segmented manner over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction to remove non curcuminoids including but not limited to resinous material and other medium polar impurities from the de-oiled turmeric while selectively keeping curcuminoids therein. Thus, after the first step of
25 co-solvent pumping, co-solvent pumping is repeated 2 more times after an interval ranging from 5 to 30 minutes, preferably 15 minutes till 1 hour. Specifically, the co-solvent is acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 min, preferably, 1 min at a temperature ranging

between 32 to 150 °C, preferably between 37 to 70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100 to 150 bars, more preferably between 130 to 180 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric. However, it is understood that the co-solvent can also be
5 selected from any of the common organic solvents such as acetone, ethyl acetate, methanol, ethanol, isopropyl alcohol and the like or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% as per various alternate embodiments of the process (200). Similarly, it is understood that the co solvent can be pumped in a continuous manner in which case the percentage of
10 co-solvent may range from 1 to 40 % of the turmeric quantum and for a period ranging from 2 minutes to 2 hours depending upon the quantum of curcuminoids present in the feed as well as on the flow rate of supercritical fluid.

At step (140), the process (200) involves solvent extraction wherein the remaining
15 residue from the product of step (130) is extracted in solvent to obtain an extract containing curcuminoids for further processing. In an embodiment, the solvent is preferably acetic acid is used at a concentration of 1 to 10 times by weight, preferably 5 times at temperature ranging from 35 to 80 °C, preferably 60 °C under stirring for a duration of 15 minutes to 2 hours, preferably 30 minutes or for a duration of 30
20 minutes to 2 hours, preferably 1 hour. However, other solvents such as hexane, ether, ethyl acetate, acetone, methanol, ethanol, and isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%, can also be used in alternate embodiments of the process (200).

25 At step (150), the process (200) involves optionally repeating the step (140) 2-3 times, thereafter combining the extracts, filtering and concentrating the extracts under vacuum at lower temperatures, preferably 40 °C.

At step (160), the process (200) involves pouring/adding concentrated extract obtained from step (150) in any one of cold water and hot water as a continuous flowing stream to precipitate curcuminoids and to remove water soluble impurities. The temperature of the water ranges from 0 to 100 °C, preferably between 15 °C to 5 70 °C. The process (200) is such that precipitation under colder or hotter conditions would lead to curcumin in powder form which can be in nano or micro sizes.

At step (170), the process (200) involves separating the precipitated curcuminoids for example by filtering under a filter press or centrifuging the precipitated curcuminoids 10 and washing repeatedly with any one of fresh cold water and hot water as a continuous flowing stream at a temperature of 15 °C to obtain partially purified curcuminoids.

At step (180), the process (200) involves drying the partially purified curcuminoids 15 using conventional drying methods and thereafter putting in an extractor.

At step (182), the process (200) involves pumping co-solvent such as acetic acid with supercritical fluid such as supercritical CO₂ over the dried curcuminoids in three steps to precipitate almost pure curcuminoids in the extractor. However, it is 20 understood that other co-solvents such as hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% can also be used in other embodiments of the process (200).

25 At step (184), the process (200) involves washing the pure curcuminoids for example with cold acetic acid at a temperature ranging between 18-30 °C to finally obtain totally solvent and heavy metal free dried curcuminoids. In an embodiment, the

totally solvent free curcuminoids are obtained in nanoparticle forms with 95 % purity. The process (200) ends at step (190).

In a third embodiment, the present invention provides the process (300) for extraction
5 of curcuminoids and specifically, the process (300) for extraction of solvent and heavy metal free curcuminoids from finely powdered turmeric as a raw material which is described in detailed step-wise manner herein below:

The process (300) begins at step (210). At step (220), the process (300) involves
10 supercritical fluid extraction which involves treating the finely powdered turmeric (herein after 'the turmeric') with supercritical fluid to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from but not limiting to supercritical CO₂, propane, butane, R22, R134, and N₂O alone or in various combinations thereof. Specifically, the turmeric is treated with supercritical CO₂ at a
15 temperature ranging between 35 to 150 °C, preferably between 37-70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100-500 bars, more preferably between 130 to 180 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric. Optionally, step (220) can also be accomplished by using co solvents such as hexane, ether, ethyl acetate, acetone,
20 acetic acid, methanol, ethanol, isopropyl alcohol and the like or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% in combination with supercritical fluids as per various alternate embodiments of the process (300).

25 At step (230), the process (300) involves extracting the de-oiled turmeric powder with a solvent. In an embodiment, the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, preferably 5 times at temperature ranging from 35 to 80 °C, preferably 60 °C under stirring for a duration of 15 minutes to 2

hours, preferably 30 minutes or for a duration of 30 minutes to 2 hours, preferably 1 hour. However, other solvents such as hexane, ether, ethyl acetate, acetone, methanol, ethanol, and isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% can also be used in alternate
5 embodiments of the process (300).

At step (240), the process (300) involves optionally repeating the step (230) 2-3 times, thereafter combining the extracts, filtering and concentrating the extracts under vacuum at lower temperatures, preferably 40 °C.
10

At step (250), the process (300) involves adding the concentrated extract to water to precipitate the curcuminoids along with impurities such as non curcuminoids including but not limited to resinous material and other medium polar impurities.

15 At step (260), the process (300) involves filtering the precipitated curcuminoids and impurities and washing repeatedly with any one of fresh cold water and hot water as a continuous flowing stream at a temperature of 15 °C to obtain partially purified curcuminoids.

20 At step (270), the process (300) involves drying the partially purified curcuminoids using conventional drying methods and thereafter putting in the extractor.

At step (280), the process (300) involves pumping a co-solvent such as acetic acid as along with supercritical fluid such as supercritical CO₂ over the dried curcuminoids
25 in three steps to precipitate almost pure curcuminoids in the extractor.

At step (285), the process (300) involves washing the almost pure curcuminoids for example with cold acetic acid to finally obtain a totally solvent and heavy metal free

dried curcuminoids. In an embodiment, the totally solvent and heavy metal free dried curcuminoids are obtained in nanoparticle forms with 95 % purity. The process (100) ends at step (90). The process (300) ends at step (290).

- 5 In a fourth embodiment, the present invention provides the process (400) for extraction of curcuminoids and specifically, the process (400) for extraction of solvent and heavy metal free curcuminoids from finely powdered turmeric as a raw material which is described in detailed step-wise manner herein below:
- 10 The process (400) begins at step (310). At step (320), the process (400) involves supercritical fluid extraction which involves treating the finely powdered turmeric (herein after 'the turmeric') with supercritical fluid to remove essential oils therefrom and obtain de-oiled turmeric. The supercritical fluid is selected from but not limiting to supercritical CO₂, propane, butane, R22, R134, and N₂O alone or in various
- 15 combinations thereof. Specifically, the turmeric is treated with supercritical CO₂ at a temperature ranging between 35 to 150 °C, preferably between 37-70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100-500 bars, more preferably between 130 to 180 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric. Optionally, step (320) can also be
- 20 accomplished by using co solvents such as hexane, ether, ethyl acetate, acetone, acetic acid, methanol, ethanol, isopropyl alcohol and the like or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% in combination with supercritical fluids as per various alternate embodiments of the process (400).
- 25 At step (330), the process (400) involves pumping the co-solvent in a periodic/segmented manner in three steps over the de-oiled turmeric while continuing the step of supercritical fluid extraction to remove non curcuminoids including but

not limited to resinous material and other medium polar impurities from the de-oiled turmeric while selectively keeping curcuminoids therein. Thus, after the first step of co-solvent pumping, co-solvent pumping is repeated 2 more times after an interval ranging from 5 to 30 minutes, preferably 15 minutes till 1 hour. Specifically, the co-solvent is acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 min, preferably, 1 min at a temperature ranging between 32 to 150 °C, preferably between 37 to 70 °C, more preferably between 40 to 55 °C, pressure ranging from 80-900 bars, preferably ranging from 100 to 150 bars, more preferably between 130 to 180 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric. However, it is understood that the co-solvent can also be selected from any of the common organic solvents such as acetone, ethyl acetate, methanol, ethanol, isopropyl alcohol and the like or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50% as per various alternate embodiments of the process (400). Similarly, it is understood that the co solvent can be pumped in a continuous manner in which case the percentage of co-solvent may range from 1 to 40 % of the turmeric quantum and for a period ranging from 2 minutes to 2 hours depending upon the quantum of curcuminoids present in the feed as well as on the flow rate of supercritical fluid.

At step (340), the process (400) involves solvent extraction wherein the remaining residue from the product of step (330) is extracted in solvent to obtain an extract containing curcuminoids for further processing. In an embodiment, the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, preferably 5 times at temperature ranging from 35 to 80 °C, preferably 60 °C under stirring for a duration of 15 minutes to 2 hours, preferably 30 minutes or for a duration of 30 minutes to 2 hours, preferably 1 hour. However, other solvents such as hexane, ether, ethyl acetate, acetone, methanol, ethanol, and isopropyl alcohol or a combination of

water with the above mentioned solvents, wherein water concentration can be up to 50% can also be used in alternate embodiments of the process (400).

At step (350), the process (400) involves optionally repeating the step (40) 2-3 times,
5 thereafter combining the extracts, filtering and concentrating the extracts under vacuum at lower temperatures, preferably 40 °C to result at the end the extract with acetic acid ranging from 5 to 99.5 % of the total mass.

At step (360), the process (400) involves re-extracting the concentrated extract
10 obtained from step (350) with supercritical fluid such as supercritical CO₂ in the extractor at temperatures between 35 to 150 °C, preferably 50 °C and pressure ranging from 80-700 bars, preferably 150 bars at a flow rate ranging from 10 to 250 kg/kg of turmeric to remove non curcuminoids and acetic acid and precipitating almost pure curcuminoids. In the case of acetic acid being 99.5 to 80 % of the total
15 mass, treatment with the supercritical CO₂ may result in curcuminoids precipitating in the nano sized form while simultaneously getting rid of non curcuminoids and acetic acid.

At step (370), the process (400) involves washing the precipitated almost pure
20 curcuminoids with cold acetic acid to obtain almost pure curcuminoids.

At step (380), the process (400) involves drying the almost pure curcuminoids using conventional drying methods to obtain a totally solvent and heavy metal free dried curcuminoids. In an embodiment, the totally solvent and heavy metal free
25 curcuminoids are obtained in nanoparticle forms with 95 % purity. The process (400) ends at step (390).

Besides extracting solvent and heavy metal free curcumin from turmeric, the processes (100), (200), (300) and (400) can also be used for preparation of solvent and heavy metal free curcumin from commercially available curcumin as per an alternate embodiment thereof. The processes (100), (200), (300) and (400) involve
5 following steps:

- Step 1: Re-dissolving commercially available curcumin containing harmful solvent traces in acetic acid from 1 to 10 times the weight of curcumin, preferably 3 to 6 times, more preferably 5 times at temperature ranging from 25 to 90 °C, preferably between 35 to 75 °C, more preferably at 50 °C.
- 10 • Step 2: Re-precipitating the solution in cold or hot water or using supercritical fluid such as supercritical CO₂ with temperature ranging from 0 to 100 °C, preferably at 15 or 65 °C to render it completely free of harmful solvents.
- Step 3: Filtering and drying curcumin using conventional methods to obtain solvent free curcumin.

15

Examples

The process of the present invention will be described in more details below by means of an example. The following example is provided in order to demonstrate and further illustrate the embodiments and aspects of the present invention and is not to
20 be construed as limiting the scope thereof.

Example 1 for the first embodiment (process (100)):

Finely powdered turmeric, 3 kg, was extracted with supercritical CO₂ at 45 °C and 150 bars at a flow rate of 40 kg/kg of turmeric for 1 hour. To the turmeric, now de-
25 oiled, acetic acid, total 300 mL, was pumped as co-solvent (with supercritical CO₂) in three steps. At each step 100 mL acetic acid was pumped over a period of 1 minute. This step was repeated 2 more times. The acetic acid treated turmeric powder was

then extracted with 9 L of acetic acid at 60 °C for 30 minutes. The acetic acid was filtered and concentrated under vacuum to 500 mL volume. The concentrated extract was then allowed to crystallize curcuminoids at 20 °C. The crystallized curcuminoids were then filtered and washed with cold acetic acid at a temperature of 20 °C. The
5 curcuminoids were dried under hot air to obtain totally solvent and heavy metal free dried curcuminoids, 75 g containing, 95 % purity.

Example 2 for the second embodiment (process (200)):

Finely powdered turmeric, 3 kg, was extracted with supercritical CO₂ at 45 °C and
10 150 bars at a flow rate of 40 kg/kg of turmeric for 1 hour. To the turmeric, now de-oiled, acetic acid, total 300 mL, was pumped as co-solvent (with supercritical CO₂) in three steps. At each step 100 mL acetic acid was pumped over a period of 1 minute. This step was repeated 2 more times. The acetic acid treated turmeric powder was then extracted with 9 L of acetic acid at 60 °C for 30 minutes. The acetic acid was
15 filtered and concentrated under vacuum to 500 mL volume. The concentrated extract was then added to water, 5 L at 15 °C. The precipitated curcuminoids were then filtered and washed repeatedly with 500 mL fresh cold water at a temperature of 15 °C. The partially purified curcuminoids were air dried and put in the extractor. Acetic acid, total 300 mL, was pumped as co-solvent (with supercritical CO₂) in three steps.
20 At each step 100 mL acetic acid was pumped over a period of 1 minute. This step was repeated 2 more times to precipitate almost pure curcuminoids in the extractor. They were then washed with cold acetic acid to finally obtain and heavy metal curcuminoids 95 % pure, 75 g.

25 **Example 3 for the third embodiment (process (300)):**

Finely powdered turmeric, 3 kg, was extracted with supercritical CO₂ at 45 °C and 150 bars at a flow rate of 40 kg/kg of turmeric for 1 hour. The de-oiled turmeric powder was then extracted with 9 L of acetic acid at 60 °C for 30 5 minutes. The

acetic acid was filtered and concentrated under vacuum to 500 mL volume. The concentrated extract was then added to water, 5 L at 15 °C. The precipitated curcuminoids were then filtered and washed repeatedly with 500 mL fresh cold water at a temperature of 15 °C. The partially purified curcuminoids were air dried and put
5 in the extractor. Acetic acid, total 300 mL, was pumped as co-solvent (with supercritical CO₂) in three steps. At each step 100 mL acetic acid was pumped over a period of 1 minute. This step was repeated 2 more times to precipitate almost pure curcuminoids in the extractor. They were then washed with cold acetic acid to finally obtain and heavy metal curcuminoids 95 % pure, 75 g.

10

Example 4 for the fourth embodiment (process (400)):

Finely powdered turmeric, 3 kg, was extracted with supercritical CO₂ at 45 °C and 150 bars at a flow rate of 40 kg/kg of turmeric for 1 hour. To the turmeric, now de-oiled, acetic acid, total 300 mL, was pumped as co-solvent (with supercritical CO₂) in
15 three steps. At each step 100 mL acetic acid was pumped over a period of 1 minute. This step was repeated 2 more times. The acetic acid treated turmeric powder was then extracted with 9 L of acetic acid at 60 °C for 30 minutes. The acetic acid was filtered and concentrated under vacuum to 500 mL volume. The concentrated extract was then fed to the extractor and subjected to extraction with supercritical CO₂ at 45
20 °C and 150 bars to remove non curcuminoids and acetic acid leaving curcuminoids 95% pure in the extractor. They were finally washed with cold acetic acid and dried to obtain and heavy metal curcuminoids 95 % pure, 75 g.

Advantages of the invention

25

1. Each process (100), (200), (300) and (400) uses green solvents that are used as food additives since time immemorial. CO₂ in the supercritical phase is a

green solvent that leaves no trace in the extract after conversion to the gaseous state.

2. Each process (100), (200), (300) and (400) uses acetic acid that has a significant effect on the acceptability by regulatory authorities and consumers alike.
3. Each process (100), (200), (300) and (400) involves acetic acid dissolution and re-precipitation in water that removes all previous traces of organic solvents and heavy metals from the extracted curcuminoids.

10 The foregoing objects of the invention are accomplished and the problems and shortcomings associated with prior art techniques and approaches are overcome by the present invention described in the present embodiment. Detailed descriptions of the preferred embodiment are provided herein; however, it is to be understood that the present invention may be embodied in various forms. Therefore, specific details
15 disclosed herein are not to be interpreted as limiting, but rather as a basis for the claims and as a representative basis for teaching one skilled in the art to employ the present invention in virtually any appropriately detailed system, structure, or matter. The embodiments of the invention as described above and the methods disclosed herein will suggest further modifications and alterations to those skilled in the art.
20 Such further modifications and alterations may be made without departing from the spirit and scope of the invention.

I CLAIM:

1. A process (100) for extraction of curcuminoids, the process (100) comprising:
 - a) treating finely powdered turmeric with any one of supercritical fluid alone
5 and supercritical fluid in combination with co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric;
 - b) pumping of co-solvent over the de-oiled turmeric in three steps while
10 continuing the step of supercritical fluid extraction at a temperature ranging between 32 to 150 °C, pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove non curcuminoids from the de-oiled turmeric while selectively keeping curcuminoids therein;
 - c) extracting remaining residue from the product of step (b) in a solvent to
15 obtain an extract containing curcuminoids, wherein the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours;
 - d) crystallizing the curcuminoids from the extract at a temperature ranging
between 18-30 °C; and
 - 20 e) filtering the crystallized curcuminoids, washing and drying to obtain totally solvent and heavy metal free dried curcuminoids.
2. The process (100) as claimed in claim 1, further comprising repeating the step (c) to combine, filter and concentrate resulting extracts.

25

3. The process (100) as claimed in claim 1, wherein the supercritical fluid used in step (a) is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.
- 5 4. The process (100) as claimed in claim 1, wherein the co-solvent used in step (a) and step (b) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%, the co-solvent is preferably acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.
- 10
5. The process (100) as claimed in claim 1, wherein the solvent used in step (c) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%.
- 15
6. The process (100) as claimed in claim 1, wherein the washing at step (e) is done with cold acetic acid at a temperature ranging between 18-30 °C.
- 20 7. A process (200) for extraction of curcuminoids, the process (200) comprising:
- a) treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric;
- 25

b) pumping of co-solvent over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction at a temperature ranging between 32 to 150 °C, pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove non curcuminoids from the de-oiled turmeric
5 while selectively keeping curcuminoids therein;

c) extracting remaining residue from the product of step (b) in a solvent to obtain an extract containing curcuminoids, wherein the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours;

10 d) adding extract obtained from step (c) in any one of cold water and hot water as a continuous flowing stream to precipitate curcuminoids and to remove water soluble impurities therefrom at a temperature ranging from 0 to 100 °C;

e) separating the precipitated curcuminoids and washing repeatedly with any one of cold water and hot water as a continuous flowing stream to obtain partially
15 purified curcuminoids;

f) drying the partially purified curcuminoids and thereafter putting in an extractor;

g) pumping co-solvent with supercritical fluid over the dried curcuminoids in three steps to precipitate almost pure curcuminoids in the extractor; and

20 h) washing the pure curcuminoids to finally obtain totally solvent and heavy metal free dried curcuminoids.

8. The process (200) as claimed in claim 1, further comprising repeating the step (c) to combine, filter and concentrate resulting extracts.

25

9. The process (200) as claimed in claim 1, wherein the solvent used in step (c) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%.

5

10. The process (200) as claimed in claim 1, wherein the supercritical fluid used in step (a) and (g) is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

10

11. The process (200) as claimed in claim 1, wherein the co-solvent used in step (a), (b) and (g) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%, the co-solvent is preferably acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.

15

12. The process (200) as claimed in claim 1, wherein the washing at step (h) is done with cold acetic acid at a temperature ranging between 18-30 °C.

20

13. A process (300) for extraction of curcuminoids, the process (300) comprising:

a) treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric;

25

- b) extracting the de-oiled turmeric with solvent, wherein the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours;
- 5 c) adding extract obtained from step (b) to water to precipitate the curcuminoids along with impurities such as non curcuminoids;
- d) filtering the precipitated curcuminoids and impurities and washing repeatedly with any one of cold water and hot water as a continuous flowing stream to obtain partially purified curcuminoids;
- 10 e) drying the partially purified curcuminoids and thereafter putting in an extractor;
- f) pumping co-solvent with supercritical fluid over the dried curcuminoids in three steps to precipitate almost pure curcuminoids in the extractor; and
- g) washing the almost pure curcuminoids to finally obtain totally solvent and
15 heavy metal free dried curcuminoids.
14. The process (300) as claimed in claim 1, further comprising repeating the step (b) to combine, filter and concentrate resulting extracts.
- 20 15. The process (300) as claimed in claim 1, wherein the solvent used in step (b) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%.

16. The process (300) as claimed in claim 1, wherein the supercritical fluid used in step (a) and (f) is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

5

17. The process (300) as claimed in claim 1, wherein the co-solvent used in step (a) and (f) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%, the co-
10 solvent is preferably acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.

18. The process (300) as claimed in claim 1, wherein the washing at step (g) is done with cold acetic acid at a temperature ranging between 18-30 °C.

15

19. A process (400) for extraction of curcuminoids, the process (400) comprising:

a) treating finely powdered turmeric with any one of supercritical fluid alone and supercritical fluid in combination with co-solvent at temperature ranging between 35 to 150 °C, at pressure ranging from 80-900 bars, and at a flow rate ranging from
20 10 to 250 kg/kg of the turmeric to remove essential oils therefrom and obtain de-oiled turmeric;

b) pumping of co-solvent over the de-oiled turmeric in three steps while continuing the step of supercritical fluid extraction at a temperature ranging between 32 to 150 °C, pressure ranging from 80-900 bars, and at a flow rate ranging from
25 to 250 kg/kg of the turmeric to remove non curcuminoids from the de-oiled turmeric while selectively keeping curcuminoids therein;

c) extracting remaining residue from the product of step (b) in solvent to obtain an extract containing curcuminoids, wherein the solvent is preferably acetic acid used at a concentration of 1 to 10 times by weight, at temperature ranging from 35 to 80 °C, under stirring for a duration of 15 minutes to 2 hours such that the
5 resulting extract having solvent preferably acetic acid ranging from 5 to 99.5 % of the total mass;

d) re-extracting the extract obtained from step (c) with supercritical fluid in an extractor at temperatures between 35 to 150 °C, pressure ranging from 80-700 bars, at a flow rate ranging from 10 to 250 kg/kg of turmeric to remove non
10 curcuminoids and acetic acid and precipitating almost pure curcuminoids;

e) washing the precipitated almost pure curcuminoids to obtain almost pure curcuminoids; and

f) drying the almost pure curcuminoids to obtain a totally solvent and heavy metal free dried curcuminoids.

15

20. The process (400) as claimed in claim 1, further comprising repeating the step (c) to combine, filter and concentrate resulting extracts.

21. The process (400) as claimed in claim 1, wherein the supercritical fluid used
20 in step (a) and step (d) is selected from any one of supercritical CO₂, propane, butane, R22, R134, N₂O and combinations thereof, the supercritical fluid is preferably supercritical CO₂.

22. The process (400) as claimed in claim 1, wherein the co-solvent used in step
25 (a) and (b) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol, or a combination of water with the

above mentioned solvents, wherein water concentration can be up to 50%, the co-solvent is preferably acetic acid pumped at a concentration of about 3% of the de-oiled turmeric over a period ranging from 0.5 to 10 minutes.

- 5 23. The process (400) as claimed in claim 1, wherein the solvent used in step (c) is selected from any one of acetic acid, hexane, ether, ethyl acetate, acetone, methanol, ethanol, isopropyl alcohol, or a combination of water with the above mentioned solvents, wherein water concentration can be up to 50%.
- 10 24. The process (400) as claimed in claim 1, wherein the washing at step (e) is done with cold acetic acid at a temperature ranging between 18-30 °C.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IN2017/050174

A. CLASSIFICATION OF SUBJECT MATTER A61K36/9066, A61K36/00 Version=2017.01		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) A61K36*		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) Patseer, IPO Internal Database		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Braga ME et al: "Comparison of yield, composition, and antioxidant activity of turmeric (Curcuma longa L.) extracts obtained using various techniques"; Journal of Agricultural and Food Chemistry; Oct 2003; Volume: 51(22); Pages: 6604-6611. See whole document especially Abstract, Page 6605 para 1, SFE Procedure, Results and Discussion	1-24
X	KR20130013686A (JEONNAM BIOINDUSTRY FOUNDATION) 06 February 2013 (06/02/2013). See Abstract	1-24
X	EP0726074A1 (A.S.A.C. PHARMACEUTICAL INTERNATIONAL, A.I.E.) 14 August 1996 (14/08/1996). Whole document, para [0018]	1-24
X	JPH069479A (KYODO KUMIAI OKINAWA PREF GOV) 18 Jan 1994 (18/01/1994). Whole document	1-24
A	Xu L et al: "Recent advances on supercritical fluid extraction of essential oils"; African Journal of Pharmacy and Pharmacology; Sep 2011;	1-24
<input checked="" type="checkbox"/>	Further documents are listed in the continuation of Box C.	<input checked="" type="checkbox"/> See patent family annex.
* Special categories of cited documents:		
"A" document defining the general state of the art which is not considered to be of particular relevance	"I" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	
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Date of the actual completion of the international search 19-09-2017	Date of mailing of the international search report 19-09-2017	
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INTERNATIONAL SEARCH REPORT

International application No.
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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Volume: 5(9); Pages: 1196-1211. Whole document	

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/IN2017/050174

Citation	Pub.Date	Family	Pub.Date
KR 20130013686 A	06-02-2013	KR 101304538 B1	05-09-2013
EP 0726074 A1	14-08-1996	DE 69531146 T2	03-06-2004
		EP 0726074 B2	25-06-2003
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