A further object of the present disclosure provides a synthetic hypericin produced by a process comprising at least one of the following steps:

(a) photoconverting protohypericin to crude hypericin using micro-reactor equipped with a Light Emitting Diode (LED) light source either as batch or continuous mode;

(b) solubilizing crude hypericin in a solvent at a pre-precipitation temperature; filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed; washing and filtering the precipitate preferably with a washing solvent; and

(c) treating crude hypericin with at least one salt forming reagent in a solvent at a pre-precipitation temperature; filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed; washing and filtering the precipitate preferably with a washing solvent, wherein the at least one step (a), (b) and (c) is followed by drying of the purified hypericin product with nitrogen, water vapor or air under vacuum.

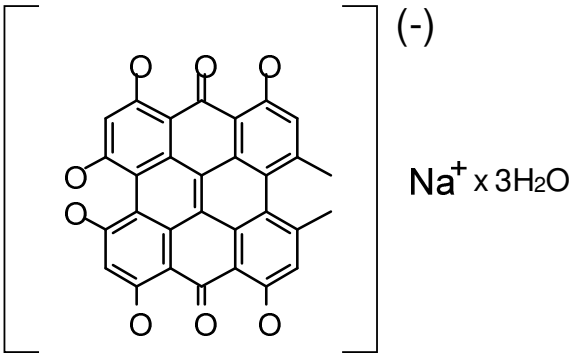
In an aspect, the present disclosure refers to a purified synthetic hypericin and hypericin derivative produced by a process comprising at least one of the following steps:

(a) photoconverting protohypericin to crude hypericin using a micro-reactor comprising a LED light source either as batch or continuous mode;

(b) solubilizing crude hypericin in a solvent at a pre-precipitation temperature, filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed and washing and filtering the precipitate in a washing solvent; and

(c) treating crude hypericin with at least one salt forming reagent in a solvent at a pre-precipitation temperature, filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed and washing and filtering the precipitate in a washing solvent,

wherein the at least one step (a), (b) and (c) is followed by drying of the purified hypericin product with nitrogen, vacuum or air under vacuum and further wherein the synthetic hypericin derivative is a mono-sodium salt in the form of a trihydrate having the following structure



and the synthetic hypericin yield is at least 75% to 100% of a dry produc.

In another example, there is provided a method of preparing a purified synthetic hypericin, the method comprising:

(a) photoconverting protohypericin to crude hypericin;

(b) treating the crude hypericin with at least one salt forming reagent in a solvent at a pre-precipitation temperature;

(c) filtering the crude hypericin from step (b) while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed;

(d) washing and filtering the precipitate from step (c) with a washing solvent; and

(e) drying of the product of step (d) with nitrogen, water vapor or air under vacuum.

In another example, there is provided a synthetically derived trihydrated hypericin salt: wherein the salt is selected from the group consisting of ammonium, calcium and magnesium. In another example, there is provided a synthetically derived hypericin salt: wherein the salt is selected from the group consisting of ammonium, calcium and magnesium.

In another example, there is provided a method of preparing a purified synthetic hypericin, the method comprising: (a) treating a purehypericin with at least one salt forming reagent in a solvent at a pre-precipitation temperature; (b) filtering the product from step (a) while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed;

(c) washing and filtering the precipitate from step (b) with a washing solvent; and (d) drying of the product of step (c) with nitrogen, water vapor or air under vacuum.

Yet another object of the present disclosure provides a method of preparing a purified synthetic hypericin wherein the irradiation is the result of exposure to standard light. Yet another object of the present disclosure provides a method of preparing a purified synthetic hypericin wherein the irradiation is the result of exposure to light. As light sources one can select suitable lamps such as a low or medium pressure mercury lamp. More preferred light sources are LED lights which can provide irradiation with a narrow desirable wavelength distribution.

Yet another object of the present disclosure provides a method of preparing a purified synthetic hypericin wherein the irradiation step is performed using a continuous flow

reactor. In a preferred example, the irradiation step is performed using a continuous flow microreactor.

Still another object of the present disclosure provides a method of preparing a purified synthetic hypericin resulting from LED irradiation, wherein the LED light has a wavelength between 350-700 nm.

Still another object of the present disclosure provides a process for manufacturing a purified synthetic hypericin.

Another object of the present disclosure provides a process for manufacturing purified synthetic hypericin wherein the synthetic hypericin yield is at least 30% of the dry product. In a preferred example the synthetic hypericin yield is at least 50% of the dry product. In a more

preferred example the synthetic hypericin yield is at least 70% of the dry product. In a most preferred example the synthetic hypericin yield is at least 75 to 100% of the dry product.

Another object of the present disclosure provides a process for manufacturing purified synthetic hypericin wherein the synthetic hypericin purity is at least 80% of the dry product. In a preferred example the synthetic hypericin purity is at least 90% of the dry product. In a more preferred example the synthetic hypericin purity is at least 95% of the dry product. In a most preferred example the synthetic hypericin purity is at least 97% of the dry product.

BRIEF DESCRIPTION OF THE FIGURES

The novel features of this invention, as well as the invention itself, both as to its structure, reaction scheme and its operation, will be best understood from the accompanying drawings, taken in conjunction with the accompanying description, in which similar reference characters refer to similar parts, and in which:

Figure 1 depicts a reaction scheme for hypericin manufacture.

Figure 2 depicts a process flow diagram for the manufacture of hypericin.

DETAILED DESCRIPTION OF THE INVENTION

The system of the present invention contains one or more of the following steps:

(1) irradiation of protohypericin to crude hypericin; (2) purification of crude hypericin in methanol – method 1; (3) purification of crude hypericin in methanol with sodium bicarbonate – method 2;

CLAIMS

What is claimed is:

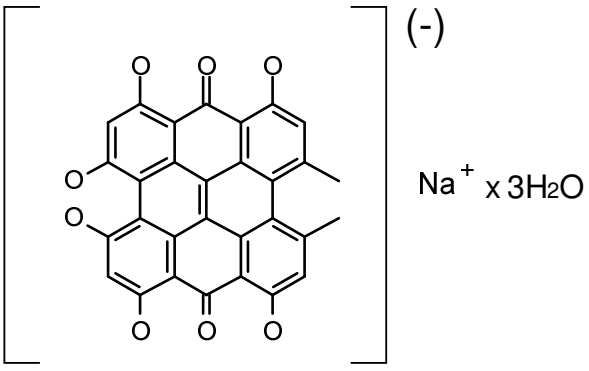
1. A purified synthetic hypericin and hypericin derivative produced by a process comprising at least one of the following steps:

(a) photoconverting protohypericin to crude hypericin using a micro-reactor comprising a LED light source either as batch or continuous mode;

(b) solubilizing crude hypericin in a solvent at a pre-precipitation temperature, filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed and washing and filtering the precipitate in a washing solvent; and

(c) treating crude hypericin with at least one salt forming reagent in a solvent at a pre-precipitation temperature, filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed and washing and filtering the precipitate in a washing solvent,

wherein the at least one step (a), (b) and (c) is followed by drying of the purified hypericin product with nitrogen, vacuum or air under vacuum and further wherein the synthetic hypericin derivative is a mono-sodium salt in the form of a trihydrate having the following structure



and the synthetic hypericin yield is at least 75% to 100% of a dry product.

2. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the solvent is selected from the group consisting of methanol, acetone, methyl ethyl ketone and ethyl acetate.

3. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the solvent from step (b) and step (c) are the same solvent.

4. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the solvent from step (b) is different from the solvent of step (c).

5. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the washing solvent is selected from at least one of the group consisting of ethyl acetate, hexane, pentane, ether and methyl tertiary butyl ether (MTBE).

6. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the at least one salt forming reagent is selected from the group consisting of sodium bicarbonate, ammonium bicarbonate, potassium bicarbonate, calcium bicarbonate, and magnesium bicarbonate.

7. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the process comprises:

photoconverting protohypericin to hypericin using a micro-reactor comprising a LED light source either as batch or continuous mode; and

drying of the resulting purified hypericin product with nitrogen, vacuum or air under vacuum.

8. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the process comprises:

solubilizing hypericin in a solvent at a pre-precipitation temperature;

filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed;

washing and filtering the precipitate preferably with a washing solvent; and

drying of the resulting purified hypericin product with nitrogen, water vapor or air under vacuum.

9. The purified synthetic hypericin and hypericin derivative of claim 1, wherein the process comprises:

treating hypericin with at least one salt forming reagent in a solvent at a pre-precipitation temperature;

filtering the treated hypericin while providing at least one cooling ramp to reach a post-precipitation temperature, wherein a precipitate is formed;

washing and filtering the precipitate preferably with a washing solvent; and

drying of the resulting purified hypericin product with nitrogen, vacuum or air under vacuum.

10. The synthetic hypericin and hypericin derivative of claim 1, wherein the process comprises: photoconverting resulting from exposure to a standard light at a wavelength between 350-700 nm.

11. The synthetic hypericin and hypericin derivative of claim 1, wherein the process comprises a pre-precipitation temperature greater than 40 ºC.

12. The synthetic hypericin and hypericin derivative of claim 1, wherein the process comprises: a post-precipitation temperature less than 40 ºC.