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(54) Title: A PROCESS FOR THE ENCAPSULATION OF GARCINIA EXTRACT

(57) Abstract: The present invention relates to a novel process for the encapsulation of *Garcinia* extract powder, which comprises (a) collecting the fruits from the species of *Garcinia*, (b) cutting the rinds of the fruits manually, (c) extracting with de-ionized water at a volume ratio of 1:4 for a period of 15-35 min at 110-130°C, (d) filtering the extract using filter cloth, (e) mixing of the *Garcinia* extract with 5-20 % (w/w) whey protein and (f) spray drying the above mixture to obtain the product in powder form.



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## A PROCESS FOR THE ENCAPSULATION OF *GARCINIA* EXTRACT

### Field of the invention

The present invention relates to a process for the encapsulation of *Garcinia* extract. The present invention particularly relates to a process of obtaining *Garcinia* extract in powder form by encapsulation using whey protein.

### Background and Prior art references

As dietary supplement, (-)-Hydroxycitric acid (HCA) is an effective addition to any weight management program. Allison et al. (Crit.Rev.Food Sci.Nutr.2001, 41,1-28) has reviewed the use of HCA as one of the alternative treatments for weight loss. The derivatives of HCA have been incorporated into many pharmaceutical preparations in combination with other ingredients for the purpose of enhancing weight loss, cardioprotection, correcting the conditions of lipid abnormalities and endurance (Jena et al., 2002, J. Agric. Food Chemistry, 50,10-22). So far, HCA has been found in the fruits of certain species of *Garcinia*, which includes *G. cambogia*, *G. indica*, *G.atroviridis* and *G. cowa* (Lewis, Y.S. 1969, Methods in enzymology, 13, 613-623; Jena et al., 2002, J. Agric. Food Chemistry, 50,10-22). The chemistry and biochemistry of HCA has been discussed recently (Jena et. al., 2002, J. Agric. Food Chemistry, 50,10-22). During extensive animal studies, HCA has been proven to effectively curb appetite, suppress food intake, increase the rates of hepatic glycogen synthesis, reduce fatty acid synthesis and lipogenesis and decrease body-weight gain.

*Garcinia* (Family: *Guttiferae*) is a large genus of polygamous trees or shrubs, distributed in the tropical Asia, Africa and Polynesia. It consists of 180 species, out of which about 30 species are found in India. *G. pedunculata* and *G. cowa* are grown in Northeastern parts of India and Andaman Islands. In Assam, *G. cowa* is often cultivated in homesteads for its acid fruits (The Wealth of India, 1956). The fruits from both the species of *Garcinia* are not palatable due to their strong acid taste. In Assam, the sun-dried slices of the fruits are used for culinary purposes and as folk medicine.

Reference may be made to the commercial samples of *Garcinia cambogia* extracts, where the HCA is present as its calcium salt (Sawada, et al, 1997, *Nihon yukagaka kaishi*, vol, 1467-1474). However, the excess calcium reduces the solubility and subsequently bioavailability when it is compared to the liquid extract.

Another reference may be made to the Ashok kumar, Ravindranath, B., and Balasubramamanvam (US patent # 656314, 1996). This process involves water extraction, followed by passing through ion exchange resins and decolorisation by using activated

charcoal and concentration. The main drawback of this method is the process involves too many unit operations for obtaining the final product.

Reference may be made to Majeed et al. (Majeed, M., Badmaev, V and Rajendran, R. US Patent. no 5,783,603, 1998), wherein the preparation of potassium hydroxycitrate from *Garcinia* fruit was reported. It involves the extraction of *Garcinia* fruit using alkyl alcohol, the extract was treated with potassium hydroxide and refluxed to form potassium hydroxycitrate precipitate. The main drawback of this method is the potassium salt is hygroscopic. Further, HCA is not available in natural state, limiting its bioavailability and versatile applicability for pharmaceutical purposes.

It may be noted that all these references are mentioned above HCA derivative preparations. However, there is no report on the encapsulation of HCA in its native (not as derivatives) form.

### **Objects of the invention**

The main object of the present invention is to develop a novel process for the encapsulation of *Garcinia* extract.

Another object of the present invention is to develop a process for the encapsulation of HCA in powder form with whey protein to get stable and non-hygroscopic.

Still another object of present invention is to get encapsulated HCA powder.

### **Summary of the Invention**

Accordingly, the present invention provides a novel process for the encapsulation of *Garcinia* extract powder, which comprises (a) collecting the fruits from the species of *Garcinia*, (b) cutting the rinds of the fruits manually, (c) extracting with de-ionized water at a volume ratio of 1:4 for a period of 15-35 min at 110-130°C, (d) filtering the extract using filter cloth, (e) mixing of the *Garcinia* extract with 5-20 % (w/w) whey protein and (f) spray drying the above mixture to obtain the product in powder form.

### **Detailed Description of the Invention**

In accordance to the objectives of the present invention, the process of encapsulation of *Garcinia* extract comprising steps of:

- a) collecting the fruits of *Garcinia pedunculata*/ *Garcinia cowa*,
- b) cutting rind fruits of the plant of step (a),
- c) extracting the cut rind fruits of step (b) with de-ionized water for a time period of 20 to 30 minutes at a temperature range of 100-135° C,

- d) filtering the water extract of step (c) through a filter cloth to obtain a water extract,
- e) mixing the water extract of step (d) with Whey protein, and
- f) spray drying the mixture of step (e) to obtain the encapsulated Garcinia extract powder.

5 In an embodiment of the invention provides a process, wherein in step (b) the size of the cut rind fruits ranges between 5 x 7 mm to 9 x 11 mm.

One more embodiment, the ratio of the plant to de-ionized water used, is in the range of 1:4 and the extraction is performed at a temperature in the range of 115°C to 125°C.

10 Another embodiment, wherein in step (e) the weight/weight ratio of water extract of Garcinia: Whey protein is in the range of 85:15 to 90:10.

Still another embodiment, the spray drying of the water extract is performed maintaining an inlet temperature in the range of 140 to 150° C and an outlet temperature in the range of 90° to 95° C.

15 Still another embodiment, the feed rate of water extract is in the range of 80 ml to 90 ml per minute.

Yet another embodiment, the hydroxycitric acid is obtained from the aqueous extract of Garcinia pedunculata or Garcinia Cowa.

20 Yet another embodiment, the hydroxycitric content of encapsulated powder of extract is in the range of 53-62% on dry basis.

Yet another embodiment, the Whey protein content of the encapsulated powder of extract is in the range of 30 – 44 % determined by Bradford method.

25 One more embodiment of the invention provides a process in which the encapsulated powder of Garcinia extract contains hydroxycitric acid in the native form and it is stable and non-hygroscopic.

Another embodiment, the encapsulated powder of Garcinia extract possesses better bioavailability. It also provides essential amino acids.

30 In the present invention, rinds of *G. pedunculata* / *G.cowa* were cut into small pieces and extracted with de-ionized water. The above extract was filtered through filter cloth. The entire filtrate was mixed with whey protein and fed to the spray dryer with a known feed rate. Spray dryer was maintained at desired inlet and outlet temperatures. Powder was collected from the bottom of the dryer in an aluminum pack and immediately sealed.

The purity of the preparation was analyzed by HPLC as described by Jayaprakasha, G.K. and Sakariah, K.K. (J. Liquid Chromatography & Related Technologies, 23, 915-923, 2000). Encapsulated hydroxycitric acid powder (0.1g) quantity was dissolved in 5ml water and made up to 100ml with water and filtered. The high performance liquid chromatographic system consisted of a Hewlett Packard HPLC model HP 1100 Series (Hewlett-Packard, CA, USA), fitted with a Waters  $\mu$ -Bondapack<sup>TM</sup> (Waters Corporation, Milford, MA, USA) C<sub>18</sub> column (250 × 4.6 mm I.D). The injection system (Rheodyne) used was 20  $\mu$ l sample loop. Detection was done by a HP 1100 series variable wavelength detector at wavelength of 210 nm. The elution was carried out with 8 mM sulphuric acid and flow rate was 1.0 ml/min under isocratic condition. A known volume (10 $\mu$ l) of the samples was injected on to the HPLC and the concentration of HCA was obtained directly from the peak area and by application of the dilution factor. The HCA concentration of the sample was expressed as g/100 g of sample. The purity of hydroxycitric acid was 20-23% and HCA lactone was 30-39% (w/w). Protein concentration was determined by Bradford method (1976) using coomassie brilliant blue G-250 using bovine serum albumin as standard. The protein content was found to be 30-44%.

#### **Product specification**

- (a). Color - cream white
- (b). Moisture content - 8-15 %
- (c). Free HCA - 20-23 %
- (d). HCA lactone - 30-39 %
- (e). Whey protein content – 30-44%
- (f). Solubility - Highly soluble in water and methanol
- (g). Storage Stability - Store under humid free condition

#### **The novelty of the present invention is**

1. Encapsulation of HCA is achieved in its native form and not as derivative (salt).
2. HCA powder obtained is in shelf stable, non-hygroscopic.
3. Hydroxycitrate content was 53-62 % (w/w)
4. The *Garcinia* extract powder obtained from the present process is highly soluble in water.
5. The final product with whey protein has additional health benefits from the angle of essential amino acid content.

The following examples are given by way of illustration of the present invention and should not be construed to limit the scope of the present invention.

### Example-1

Fruit rinds of *G. pedunculata* in 500g quantity were cut into small pieces and extracted with 1.5 liters of de-ionized water for a period of 20 min at 120°C. The above extract was filtered through filter cloth. The filtrate (1.5 kg) was mixed with 150g whey protein and fed through the spray dryer with the feed rate of 75ml/min. The inlet and outlet temperature of spray dryer was maintained at 135°C and 95°C. HCA Powder was collected from the bottom of the dryer in an aluminum pack and sealed immediately. The HCA content was determined by HPLC method and the hydroxycitrate content was 61% (w/w). The protein content was found to be 30%.

### Example-2

Fruit rinds of *G. cowa* in 1000g quantity were cut into small pieces and extracted with 3 liters of de-ionized water for a period of 30 min at 130 psi using autoclaving. The extract was filtered through filter cloth. The entire filtrate (3kg) was mixed with 300 g whey protein and fed through the spray dryer with the feed rate of 80ml/min. The inlet and outlet temperature of spray dryer was maintained at 150°C and 90°C, respectively. HCA powder was collected from the bottom of the dryer in an aluminum pack and sealed immediately. The HCA content was determined by HPLC method and the hydroxycitrate content was 53% (w/w). The protein content was found to be 34%.

### Example-3

Fruit rinds of *G. pedunculata* in 100gm quantity were cut into small pieces and extracting with 1liter of de-ionized water for a period of 50 min at 120°C using autoclaving. The extract was filtered through filter cloth. The entire filtrate (1-kg) was mixed with 15g of whey protein and fed through the spray dryer with the feed rate of 95 ml/min. The inlet and outlet temperature of spray dryer was maintained at 155°C and 94°C, respectively. Powder was collected from the bottom of the dryer in an aluminum pack and immediately sealed. The HCA content was determined by HPLC method and the hydroxycitrate content was 62% (w/w). The protein content was found to be 31%.

### The main advantages of the present invention are

1. This is a simple process for obtaining the encapsulated *Garcinia* powder.
2. This encapsulated of *Garcinia* powder is in its native form without being any of its derivatives like sodium, potassium and calcium salts. Hence, it will have better bioavailability.
3. Recovery of final product is high, because it is less hygroscopic.

**Claims**

1. A process for the encapsulation of Garcinia extract the said process comprising steps of:
  - a) collecting the fruits of *Garcinia pedunculata*/ *Garcinia cowa*,
  - 5 b) cutting rind fruits of the plant of step (a),
  - c) extracting the cut rind fruits of step (b) with de-ionized water for a time period of 20 to 30 minutes at a temperature range of 100-135° C,
  - d) filtering the water extract of step (c) through a filter cloth to obtain a water extract,
  - 10 e) mixing the water extract of step (d) with Whey protein, and
  - f) spray drying the mixture of step (e) to obtain the encapsulated Garcinia extract powder.
2. A process as claimed in claim 1, wherein in step (b) the size of the cut rind fruits ranges between 5 x 7 mm to 9 x 11 mm.
- 15 3. A process as claimed in claim 1, wherein in step (c) the ratio of the plant to de-ionized water used, is in the range of 1:4.
4. A process as claimed in claim 1, wherein in step (c) the extraction is performed at a temperature in the range of 115° C to 125°C.
5. A process as claimed in claim 1 wherein in step (e), the weight/weight ratio of water  
20 extract of Garcinia: Whey protein is in the range of 85:15 to 90:10.
6. A process as claimed in claim 1, wherein in step (f) the spray drying of the water extract is performed maintaining an inlet temperature in the range of 140 to 150° C and an outlet temperature in the range of 90° to 95°C.
7. A process as claimed in claim 1, wherein in step (f) the feed rate of water extract is in  
25 the range of 80 ml to 90 ml per minute.
8. A process as claimed in claim 1, wherein the hydroxycitric acid is obtained from the aqueous extract of *Garcinia pedunculata* or *Garcinia Cowa*.
9. A process as claimed in claim 1, wherein the hydroxycitric content of encapsulated powder of extract is in the range of 53-62% on dry basis.
- 30 10. A process as claimed in claim 1, wherein the Whey protein content of the encapsulated powder of extract is in the range of 30 – 44 % determined by Bradford method.
11. A process as claimed in claim 1, wherein encapsulated powder of Garcinia extract contains hydroxycitric acid in the native form.

12. A process as claimed in claim 1, wherein the encapsulated powder of *Garcinia* extract is a stable and non-hygroscopic.
13. A process as claimed in claim 1 wherein the encapsulated powder of *Garcinia* extract possess better bio-availability.
- 5 14. A process as claimed in claim 1, wherein the encapsulated powder of *Garcinia* extract provides essential amino acids.



# INTERNATIONAL SEARCH REPORT

Internat	Application No
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**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 7 A23L1/22 A23P1/04 A23L1/226 A23L1/226 A23L1/30

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)  
 IPC 7 A23P A23L

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 practical, search terms used)

EPO-Internal, WPI Data, PAJ, BIOSIS, FSTA

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 783 603 A (RAJENDRAN R ET AL) 21 July 1998 (1998-07-21) column 3, line 4-62 column 5, line 3-51 ---	1-14
A	US 5 536 516 A (RAVINDRANATH BHAGAVATHULA ET AL) 16 July 1996 (1996-07-16) column 2, line 24-61 examples 1-3 ---	1-14
A	US 6 160 172 A (BALASUBRAMANYAM KARANAM ET AL) 12 December 2000 (2000-12-12) column 2, line 6-18 example 1 ---	1-14
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

\* Special categories of cited documents :

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- \*E\* earlier document but published on or after the international filing date
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- \*P\* document published prior to the international filing date but later than the priority date claimed

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- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer  <b>Couzy, F</b>

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	US 5 911 992 A (AHMED AFTAB J ET AL) 15 June 1999 (1999-06-15) column 5, line 20-34,44 column 6, line 32-41 -----	1-14
A	GIBBS B F ET AL: "ENCAPSULATION IN THE FOOD INDUSTRY: A REVIEW" INTERNATIONAL JOURNAL OF FOOD SCIENCES AND NUTRITION, CARFAX PUBLISHING LTD, GB, vol. 50, no. 3, 1999, pages 213-224, XP009013399 ISSN: 0963-7486 page 218 -----	1-14

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Information on patent family members

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