

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
23 November 2006 (23.11.2006)

PCT

(10) International Publication Number
WO 2006/123363 A2

(51) International Patent Classification:
B01D 11/02 (2006.01)

(21) International Application Number:

PCT/IN2006/000087

(22) International Filing Date: 10 March 2006 (10.03.2006)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

593/MUM/2005 16 May 2005 (16.05.2005) IN

(71) Applicant and

(72) Inventor: CHAUHAN, Prabhat, Singh [IN/IN]; 602 Sabari Aangan, 11th Road, Chembur, Mumbai 400 071, Maharashtra (IN).

(74) Agent: NAIR, Gopakumar, G.; Patent & Trademark Agent, Gopakumar Nair Associates, Nair Baug, Akurli Road, Kandivli (East), Mumbai 400 101 (IN).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE,

KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

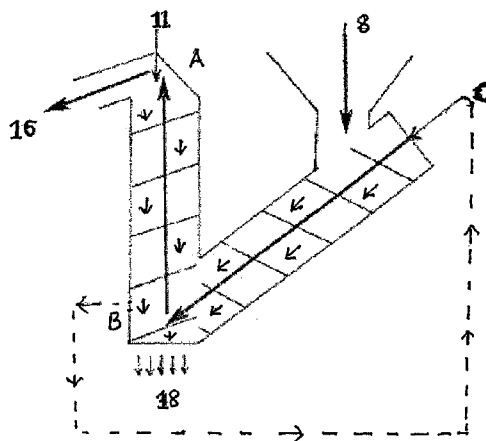
- as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))
- as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))
- of inventorship (Rule 4.17(iv))

Published:

- without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: A COUNTER-CURRENT-CUM -CONCURRENT EXTRACTOR AND THE METHOD OF EXTRACTION BY USING THE SAME



(57) Abstract: A counter-current-cum-concurrent extractor (1/20) comprises a substantially "V"-shaped body (2) having a pair of inclined tubular arms (3 and 4) interconnected at the bottom (5), the said arms forming a concurrent cylinder (C1) and a counter-current cylinder (C2), an inlet (7) for feeding the raw material (8) provided at/near the top end of the concurrent cylinder, an inlet (10) for feeding the solvent (11) and an outlet (15) for discharging the waste (16) of extracted raw material provided at/near the top end of the counter-current cylinder, an opening (22) for sucking out the extract provided near the bottom of the counter-current cylinder, an inlet (12) for feeding the particularly saturated extract (13), received from the counter-current cylinder (C2), provided near top end of the concurrent cylinder (C1), an outlet (19) for recovering the final concentrated extract (18) provided at the bottom of the extractor below the junction (5) of the said two inclined arms and means (P1, P2/S1, S2) for gradually moving the raw material from top to bottom of the concurrent cylinder and then from bottom to top of the counter-current cylinder and the said means being provided with perforations (17/21), such that the solvent and/or the extract passes downwards therethrough without allowing any raw material particles to pass through.



WO 2006/123363 A2

A COUNTER-CURRENT-CUM -CONCURRENT EXTRACTOR AND THE METHOD OF EXTRACTION BY USING THE SAME

TECHNICAL FIELD OF INVENTION:

This invention relates to a counter-current-cum-concurrent extractor and the method of extraction by using the same.

More particularly this invention relates to a counter-current-cum-concurrent extractor used for continuous extraction of soluble components from solid (powder) materials including herbs using aqueous or non aqueous solvents where the solvent is made to flow in the opposite direction (counter-current) to the direction of movement of the raw material as well as in the same direction (concurrent) to achieve maximum yield of extraction with excellent product quality at much lower costs.

BACKGROUND OF INVENTION AND PRIOR ART:

Prior art describes various types of counter-current extractors such as multi-stage extractor, horizontal worm extractor, horizontal screw extractor, vertical worm type, horizontal auger type, total wall type, centrifugal type and others.

U.S. Patent nos. 5412126 and 5132456 disclose the sorption regeneration process of carboxylic acid using concurrent extraction.

U.S. Patent no. 5320861 discloses extraction of fruits such as cranberries using counter-current extractor.

U.S. Patent no. 6228993 discloses a novel process for making an isoflavone concentrate product from soybeans involving the use of concurrent extractor in one step.

U.S. Patent no. 5635189 discloses a liquid/liquid extracting process using counter-current extractors for tocopherol.

It is seen from prior art as above that counter-current and concurrent extraction has individually been described for the extraction of materials such as vegetable drugs, fruits, tobacco, salts, oil-seeds, foods, coal and a number of other materials.

In the laboratory, and in the kilolabs, methods such as trituration, maceration, percolation and decoction are adopted to obtain extracts of solid materials, wherein the soluble portion is removed from the residue and concentrated to obtain the extracted material in solid or concentrated solution/paste form. Extraction of herb is a complicated process and can be affected by various factors such as cell wall thickness, particle size, porosity, cell structure, maceration time, isothermal behaviour of active ingredient, stability of active ingredient, diffusion rate, osmotic concentration etc. Further these factors can also affect the stability of the extract. To achieve maximum yield and a stable product, process parameters such as temperature of extracting medium, maceration time etc. have to be regulated.

There is in use an apparatus such as soxhlet extractor in laboratory practice, which is a batch type device for extraction of soluble constituents. However, such extraction facilities do not employ methods wherein continuous flow-in of solvents and raw materials is employed to obtain continuous flow-out of extracted materials.

OBJECTS OF THE INVENTION:

The main object of this invention is to provide a counter-current-cum-concurrent extractor in which continuous flow-in of solvent and raw material is employed to obtain continuous flow-out of extracted materials of excellent quality with maximum yield.

Another object of this invention is to provide a counter-current-cum-concurrent extractor which requires a very low area /floor space for its installation and uses very less man power.

A further object of this invention is to provide a counter-current-cum-concurrent extractor which is capable of dissolving total soluble constituents present in the raw material, thereby achieving 100% extraction of soluble constituents.

A further object of this invention is to provide a counter-current-cum-concurrent extractor in which maceration time, infusion time, and percolation time can be easily varied as per requirements simply by changing the speed of movement of the raw material.

SUMMARY OF THE INVENTION:

A counter-current-cum-concurrent extractor (as in fig 1 and fig 2) is described and claimed herein. The process of continuous extraction of soluble components from solid (powder) materials including herbs using aqueous or non aqueous solvents where the solvent is made to flow in the opposite direction (counter-current) to the direction of movement of the raw material as well as in the same direction (concurrent) to achieve maximum yield of extraction with excellent product quality at much lower costs is described.

DETAILED DESCRIPTION:

Accordingly this invention provides:

A counter-current-cum-concurrent extractor comprising a substantially 'V'- shaped body having a pair of inclined tubular arms interconnected at the bottom, the said arms forming a concurrent cylinder and a counter-current cylinder, an inlet for feeding the raw material provided at/near the top end of the concurrent cylinder, an inlet for feeding the solvent and an outlet for discharging the waste of extracted raw material provided at /near the top end of the counter-current cylinder, an opening for sucking out the extract provided near the bottom end of the counter-current cylinder, an inlet for feeding the extract received from the counter-current cylinder, provided near top end of the concurrent cylinder, an outlet for recovering the final concentrated extract provided at the bottom of the extractor below the junction of the said two inclined arms and means for gradually moving the raw material from top to bottom of the concurrent cylinder and then from bottom to top of the counter-current cylinder and the said means being provided with perforations, such that the solvent and / or the extract passes downwards therethrough without allowing any raw material particles to pass through.

A reciprocating piston provided in each cylinder gradually moves the raw material from top to bottom in the concurrent cylinder and pushes the raw material from bottom to top in the counter-current cylinder. The pistons are reciprocated by hydraulic, pneumatic or mechanical power preferably with speed and stroke controlling system.

A non return check valve is provided in the counter-current cylinder at the junction of the two arms.

The screw conveyors having perforated blades in each cylinder gradually moves the raw material from the top to bottom in the concurrent cylinder and from bottom to top in the counter-current cylinder. The screw conveyors are rotated by electric motor or engine provided with speed controlled system.

The inlet near the top end of the concurrent cylinder for feeding the raw material is provided with a hopper for feeding the material in the powder form.

The counter-current cylinder at the top end is provided with a hood for discharging the extracted powder waste.

The partially saturated extract may be removed using reciprocating pump, vacuum pump, or peristaltic pump at the bottom of the counter-current cylinder and feeding the same at the top end of the concurrent cylinder.

The two arms/cylinders are provided with a jacket for circulating hot water there through to control and maintain the temperature of the solvent and the raw material at a desired level needed for dissolving maximum amount of soluble constituents in the raw material.

The method of extraction by using the counter-current-cum-concurrent extractor involves the following steps:-

- i) feeding raw material, preferably in powder form from the top of the concurrent cylinder, gradually and continuously or intermittently from top to the

- bottom side of the concurrent cylinder and then from bottom to top side of the counter-current cylinder,
- ii) feeding solvent from the top of the counter-current cylinder,
 - iii) removing the partially saturated extract at the bottom side of the counter-current cylinder,
 - iv) feeding the partially saturated extract at the top side of the concurrent cylinder for maceration/wetting of the freshly entering raw material, softening the raw material, helping in flowing down of the raw material getting saturated before it reaches the bottom of the concurrent cylinder,
 - v) discharging the waste of extracted raw material, from the top side of the counter-current cylinder, and,
 - vi) collecting the saturated extract from the outlet at the bottom of concurrent cylinder/extractor.

The particle size of the raw material powder is varying in the range of 0.5mm to 5mm. The temperature of the solvent and the raw material like herb powder is controlled by circulating hot water in the jacket outside the cylinders of the extractor, to dissolve all the soluble constituents of the raw material.

DESCRIPTION OF DRAWING:

The invention will now be described with reference to the accompanying drawings wherein:

Fig 1 - shows a systematic perspective view of counter-current-cum-concurrent extractor according to the embodiment of this invention.

Fig 2 - shows a systematic perspective view of counter-current-cum-concurrent extractor according to another embodiment of this invention.

Fig 3 – shows a flow diagram of continuous extraction process/ method using the counter-current-cum-concurrent extractor of this invention.

Now referring to the Figs 1 to 3, the counter-current-cum-concurrent extractor (1) (shown in Fig 1) according to an embodiment of this invention mainly comprises a V

shaped body (2) having tubular arms/cylinder (3 and 4) interconnected with each other near the bottom end (5) of the extractor (1). The arm (3) works as a concurrent cylinder (C1) and the arm (4) works as counter-current cylinder (C2) of the Extractor (1). The cylinder or the tubular arms (3 and 4) are preferably enclosed by a jacket (6) for heating. The arm (3) is provided with a hopper (7) for feeding the solid materials (8) preferably in the powder form. The tubular arms (3 and 4) serve as cylinders (C1 and C2) for plying pistons (P1 and P2) one each, therein. A non-return check valve (9) is provided in the arm (4) at its lower end, which is a one way valve, used for allowing the powder movement upwards inside the arm (4), in its open position when pushed by the piston (P2), but prevents the downward movement of the powder/extract or solvent. A solvent inlet (10) is provided at the upper end of the arm (4) for feeding fresh solvent (11) and another inlet (12) is provided at the upper end of the arm (3) for feeding back the solvent with partially saturated extract (13), collected at the lower end of the arm (4) through an opening (22) for further saturation. A pump (14) such as peristaltic pump, reciprocating pump or vacuum pump is provided for pumping back the solvent with the extract (13) at the upper end of arm (3) a hood /outlet (15) is provided at the upper end of the arm (4) for continuously discharging extracted material waste (16) out of the extractor. Pistons (P1 and P2) are provided with perforations (17) depending upon the particle size of the raw material powder (8) such that the powder should not flow out there through, but the solvent, particularly concentrated extract and final solution /concentrated extract (18) passes out easily which is collected through an extract outlet (19) provided at the bottom of the V-shaped body (2).

The counter-current-cum-concurrent extractor (20) according to another embodiment of this invention is similar to the extractor (1) as described herein before with the difference that pistons (P1 and P2) are replaced by screw (S1 and S2) and the non-return check valve (9) is eliminated. The perforation (21), similar to the perforations (17) in the piston are provided in the blade of the screws (S1 and S2). Means for circulating hot water, through the jacket (6) at the outer side of the cylinders (C1 and C2) are provided for controlling / maintaining the desired temperature of the solvent and herb powder.

The dried herbs in powder form (size varying from 0.5mm to 5mm) are introduced from the hopper (7) that travels down the concurrent cylinder (C-1).

Different mechanisms such as perforated screw (S1) or perforated piston (P1) (driven by hydraulic, pneumatic or electric motor) are used to push the herb powder (8) down into the con-current cylinder (C1). A similar mechanism (S2/P2) as explained earlier pushes the powder (8) up in the counter-current cylinder (C2). A non return check valve (9) prevents the flow of herb powder back when the piston mechanism is used. When the perforated piston (P1) is in pushed down position, the perforated piston (P2) is in retracted position.

Screw and piston are perforated to prevent the herb powder flowing through yet allowing the solvent to pass through them.

The solvent (11) is introduced from point (A) at a constant flow rate, the solvent percolates counter-currently through the herb dissolving all the soluble ingredients and getting partially saturated. It is removed at (B) by using peristaltic pump or by using a vacuum and introduced at point (C). The solvent travels in the direction of the herb powder (concurrent flow) and get saturated, it travels to the bottom by gravity and flows out as a concentrated herb extract solution, through the outlet (19).

Since the herb powder is in constant or intermittent motion and starving solvent is introduced from top which comes in contact with depleted herb powder it dissolves what ever soluble constituents are remaining in the herb. This process gives 100% extraction of soluble constituents.

The maceration, in fusion time and percolation time can be varied by changing the speed of movement of herb powder by changing the speed of perforated screw or by changing the frequency of the piston or by changing the stroke of the piston.

Taking out the solution at point (B) and reintroducing at point (C), not only improves the flow and maceration of the herb powder; it also dissolves soluble ingredients and makes the solution saturated.

The design of screw is based on the particle size, which determines the size of perforation and swelling properties of the herb powder determines the pitch of the screw.

Similarly in case of piston type extractor, the perforations on piston depends on the particle size of the herb powder, and variable diameter cylinder dimensions are determined by swelling properties of the herb powder.

In counter-current extraction, fresh (starving) solvent is introduced from top, which comes in contact with depleting (exhausting) solid (Herb) powder moving in the opposite direction, since the solvent is fresh (not containing any dissolved constituents) it has a high capacity to dissolve and it extracts out whatever soluble constituents are remaining in the powder. The solid without any soluble constituents (solid waste) are discharged out since it is in continuous motion. As the solvent (now a solution) moves down it gets concentrated and meets new layers of solid powder containing higher amount of soluble constituents. The flow of liquid (solvent) and its temperature is controlled in such a way that the moving liquid always maintains a solubilisation potential or it has a capacity to dissolve more and more soluble constituents as it encounters powder rich in soluble constituents. The solvent (solution) gets partially saturated. At this point the partially saturated solution is removed from the counter-current cylinder and introduced in the concurrent cylinder.

This serves following purposes:

- 1) wetting (maceration) of the freshly introduced powder,
- 2) softening of herb
- 3) making the solution saturated before it is discharged,
- 4) helps in the flow of the powder.

This flow of solution down the concurrent cylinder is called concurrent flow. In this type of flow the liquid travels along with the powder and with time gets saturated. This helps in getting nearly 100% recoveries of soluble constituents from the solid (herb powder).

The extractor is capable of extracting 100gm to 5000kg of herbal drug in powder form per hour. The design of the extractor is such that by increasing the number of concurrent and counter-current cylinders (arranging them in parallel) the capacity can be increased.

The size of the counter-current cylinder varies from 50cm to 1000cm and the size of concurrent cylinder varies from 30cm to 600cm.

Comparative analysis of different extraction process

Extraction of about 200 kg per hour

Parameter	Conventional process	Batch of prior art	Continuous extraction process of invention.
Equipment	1. 500 L SS containers – 4 no. 2. Gas burners 3. Filter press 4. Hydraulic press	1. 100 L SS vessels – 2 no. 2. Vacuum pump 3. Boiler 4. Loading device	Continuous extractor
Man power/24 hour	Approx 36	Approx 12	3
Extraction yield	60-65%	70-80%	upto 100 %
Product quality	Unsatisfactory	Satisfactory	Excellent
Area required	2400 sq.ft.	1800 sq.ft	600 sq.ft

Extractors are coupled with an evaporator and a dryer to obtain semi-solid or solid product.

The above descriptions with reference to Figs. of the drawings is given to understand the invention rather than to limit its scope.

I Claim,

1. A counter-current-cum-concurrent extractor comprising a substantially 'V'-shaped body having a pair of inclined tubular arms interconnected at the bottom, the said arms forming a concurrent cylinder and a counter-current cylinder, an inlet for feeding the raw material provided at/near the top end of the concurrent cylinder, an inlet for feeding the solvent and an outlet for discharging the waste of extracted raw material provided at/near the top end of the counter-current cylinder, an opening for sucking out the extract provided near the bottom end of the counter-current cylinder, an inlet for feeding the extract received from the counter-current cylinder, provided near top end of the concurrent cylinder, an outlet for recovering the final concentrated extract provided at the bottom of the extractor below the junction of the said two inclined arms and means for gradually moving the raw material from top to bottom of the concurrent cylinder and then from bottom to top of the counter-current cylinder and the said means being provided with perforations, such that the solvent and /or extract passes downwards therethrough without allowing any raw material particles to pass through.
2. The extractor according to claim 1, wherein the said means is a reciprocating piston provided in each cylinder for gradually moving the raw material from top to bottom in the concurrent cylinder and pushing the raw material from bottom to top in the counter-current cylinder.
3. The extractor according to claim 2, wherein a non return check valve is provided in the counter-current cylinder at the junction of the two arms.
4. The extractor according to claim 1, wherein the said means is a screw conveyors having perforated blades provide in each cylinder for gradually moving the raw material from the top to bottom in the concurrent cylinder and from bottom to top in the counter-current cylinder.
5. The extractor according to claim 1, wherein the said inlet near the top end of the concurrent cylinder for feeding the raw material is provided with a hopper for feeding the material in powder form.
6. The extractor according to claim 1, wherein the said counter-current cylinder at its top end is provided with a hood for discharging the extracted powder waste.

7. The extractor according to claim 1, wherein a pump such as reciprocating pump, vacuum pump, or peristaltic pump is used for removing out the partially saturated extract at the bottom of the counter-current cylinder and feeding the same at the top end of the concurrent cylinder.
8. The extractor according to claim 1, wherein each of the said two arms/cylinders is provided with a jacket for circulating hot water therethrough to control and maintain the temperature of solvent and the raw material at a desired level needed for dissolving maximum amount of soluble constituents in the raw material.
9. The extractor according to claim 2, wherein the said pistons are reciprocated by hydraulic, pneumatic or mechanical power preferably provided with speed and stroke controlling system.
10. The extractor according to claim 4, wherein said screw conveyors are rotated by electric motor or engine provided with speed controlling system.
11. A counter-current-cum-concurrent extractor substantially as herein described and illustrated in fig 1 and 2 of drawings accompanying specification.
12. A method of extraction by using a counter-current-cum-concurrent extractor according any of the claims 1 to 11, comprising the following steps:-
 - i) feeding raw material, preferably in powder form from the top of the concurrent cylinder, gradually and continuously or intermittently from top to the bottom side of the concurrent cylinder and then from bottom to top side of the counter-current cylinder,
 - ii) feeding solvent from top of the concurrent cylinder,
 - iii) removing the partially saturated extract at the bottom side of the counter-current cylinder,
 - iv) feeding the partially saturated extract at the top side of the concurrent cylinder for maceration/wetting of the freshly entering raw material, softening the raw material, helping in flowing down of the raw material and getting saturated before it reaches the bottom of the concurrent cylinder,
 - v) discharging the waste of extracted raw material, from the top side of the counter-current cylinder, and,
 - vi) collecting the saturated extract from the outlet at the bottom of concurrent cylinder/extractor.

13. The method of extraction according to claim 12, wherein the particle size of the raw material powder is varying in the range of 0.5mm to 5mm.
14. The method of extraction according to claim 12, wherein the temperature of solvent and raw material like herb powder is controlled by circulating hot water in the jacket outside the cylinders of the extractor, to dissolve all the soluble constituents of the raw material.
15. The method of extraction by using the counter-current-cum-concurrent extractor substantially as herein described and illustrated in fig 1 to 3 of drawings accompanying specification.

Figure 2/3

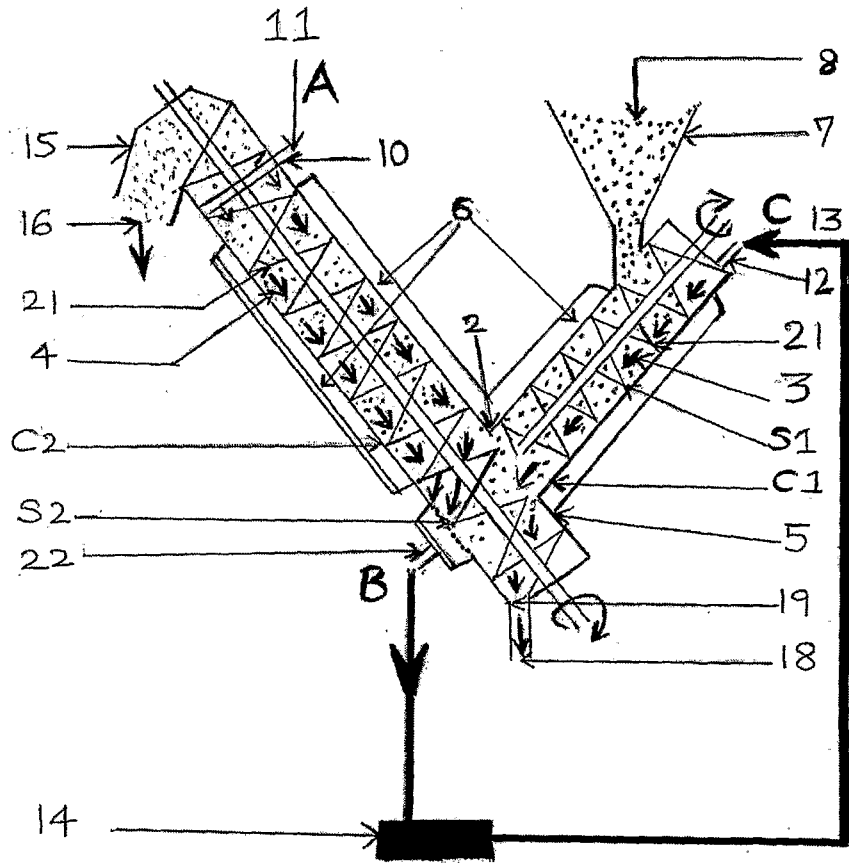


Figure 3/3

