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(54) Title: PROCESS FOR MANUFACTURING OF GREEN TEA WITH ENHANCED FLAVORS

(57) Abstract: The present invention discloses a new process for the manufacturing of high quality flavored green tea with enhanced aroma and flavor characters. The invention more particularly discloses energy efficient novel process for the manufacturing of flavored green tea under controlled temperature and pressure.

PROCESS FOR MANUFACTURING OF GREEN TEA WITH ENHANCED FLAVORS

Field of the invention

The present invention relates to an energy efficient process for manufacturing of high quality and enhanced flavored green tea. The invention more particularly relates to standardization of parameters like temperature and atmospheric pressure in order to retain majority of tea aroma and flavors, so as to enhance the overall green tea quality without any extraneous adulteration.

Background of the invention

Tea is a highly consumed refreshing beverage. It is the hot water infusion of dried and processed leaves of *Camellia sinensis* plant. Based on the type of manufacture generally there are three main types of tea; black tea, green tea and oolong tea. The green and black teas are the most frequent forms of tea consumed over worldwide. Although, there also exists few other types of teas like white tea, yellow tea and herbal teas. Black tea is further of two types- Orthodox black tea and CTC black tea. Orthodox black tea is manufactured accordingly traditional manufacture process of rolling the withered tea shoots (a bud along with upper two to three leaves) and is also known as leaf tea, while black granular CTC (crush, tear, and curl) tea is finely grounded with the help of CTC machine to get small and large granules. While oolong tea is a semifermented tea which lies between green and black tea.

Green tea is manufactured in such a way so as to kept majority of chemical constituents in made tea which are naturally present in tea shoots. This can be done by inactivating the endogenous enzymes present in the fresh tea shoots and are responsible for causing biochemical changes in tea shoots during tea processing. During green tea manufacture process, these endogenous tea enzymes were inactivated by applying heat treatment to freshly plucked tea leaves which cause denaturation of enzymes. The heat treatment for enzymes inactivation is done by various techniques, either by steaming or panning or roasting freshly plucked tea shoots. Also there exist few other ways for inactivation of tea enzymes likewise; by dipping tea leaves in boiling water or by treating green tea leaves with microwaves using microwave oven.

The green tea manufaturing (Zhen, Y. S. 2002. Tea Bioactivity and Therapeutic Potential. Taylor and Francis. London, U.K. Page no. 38) by various process methodologies involved varying range of elevated temperatures in order to inactivate the tea enzymes. During pan firing or pan roasting the temperature may reaches up to 180° C while processing of tea leaves by steaming is done at 100° C. Also few manufacturers take up temerature from 220° C to 300° C in machines for inactivating tea enzymes during processing. Therefore the green tea manufaturing by various process methodologies involve a wide range of elevated

temperatures from 100-300° C. This temerature is much higher than the evaporating temperature of various volatile components that leads to loss or degradation of numerous flavors and aroma components.

Because, during green tea manufacturing process, the enzyme inactivation techniques of fresh tea shoots as mentioned above involves usage of high temperature which thereby leads to loss of huge sum of valuable low volatile compounds of tea which are responsible for imparting various flavors and aroma. Also, drying at high temperature also results in detioration of tea aroma as well as infusion quality. The flavor and aroma are most important and critical parameters in tea quality assesment. Therefore, there is great need for some technological and scientific interventions in green tea manufacturing process so that overall flavor and aroma characteristics of made tea could be enhanced.

Earlier, Colliver et al. 2010 (US 7788364B2) disclosed that adding E-2-hexenal to a green tea product actually enhances the aroma of the green tea product. According to Colliver et al. green tea products generally have low hexenals and have aroma enriched in floral and citrus notes which thereby reduce typical grassy and green notes of green tea infusion as well as lowers the aroma quality of green tea based products. So here in this invention we have developed a novel process to manufacture green tea with high content of grassy and green notes in tea infusion without any blending.

Bagaria et al., 2009 (US 2009/0029003 A1) disclosed that drying the tea by ordinary conventional dryer leads to loss of aroma. So according to them the aroma lost during drying can be recovered which thereby can be reused to enhance the aroma of final tea product and made tea as well. Also Schutz et al., 1989 (4880656) in a US patent disclosed a process of dearomatizing and then rearomatizing of black tea and green tea by first collecting volatile flavors from tea and then adding those components through dry inert gas stream. According to them tea green and black manufactured by this methodology was more preferable thereto. But here, in the present invention, we have disclosed a new process to manufacture green tea under controlled conditions of temperature and atmospheric pressure so that the made tea retains maximum flavor and aroma compounds, higher than that of green teas made by other process methodologies and without any extraneous adulteration.

Objectives of the invention

The present invention was carried out with following objectives:

1.) To develop energy efficient process for manufacturing of green tea.

2.) To enhance the quality (flavor and aroma) of green tea without any extraneous adulteration.

3.) To manufacture natural flavored green tea by inactivating endogenous enzymes (polyphenol oxidase and peroxidase) under controlled

temperature and atmospheric pressure, so as to develop a novel process for flavored green tea manufacturing.

4.) To retain augmented desirable aroma characteristics in green tea.

5.) To have full control on enzymatic oxidation process during green tea manufacturing processing through modulation of atmospheric pressure and temperature.

The present invention is presented with the help of figures 1-8.

Figure 1. RP- HPLC chromatogram showing theanine, caffeine and catechins in new green tea (GT-A).

Figure 2. RP- HPLC chromatogram showing theanine, caffeine and catechins in green tea, processed in oven (GT -B).

Figure 3. RP- HPLC chromatogram showing theanine, caffeine and catechins in green tea, processed by steaming (GT- C).

Figure 4. RP- HPLC chromatogram showing theanine, caffeine and catechins in green tea, processed by pan roasting (GT-D).

Figure 5. GC profile of major volatile constituents present in new green tea (GT-A).

Figure 6. GC profile of major volatile constituents present in green tea, processed in oven (GT -B).

Figure 7. GC profile of major volatile constituents present in green tea, processed by steaming (GT- C).

Figure 8. GC profile of major volatile constituents present in green tea,

processed by pan roasting (GT-D).

Table 1. Data showing amount of major constituents of tea in different samples of green tea.

Table 2. Data showing major volatile constituents in different samples of green tea.

Summary of the invention

In the first aspect, the present invention provides an energy efficient process for manufacturing of naturally flavored green tea, under controlled conditions of temperature and atmospheric pressure.

In the second aspect, the present invention disclosed a new process for manufacturing of green tea by arresting endogenous tea shoot enzymes by controlled parameters of temperature and atmospheric pressure.

Accordingly the present invention provides an energy efficient process for manufacturing of novel green tea with unique and enhanced flavors which comprises the steps of:

a) withering freshly plucked tea shoots at controlled temperature and pressure for 2-3 hours , wherein temperature is in the range of 60 - 70° C and atmospheric pressure is in the range of 0.6 - 0.7 atm,

b) rolling the treated tea (controlled withered) under controlled condition for specific time period ranging 10 -15 minutes

c) drying of rolled tea at controlled temperature and pressure, at temperature range of 60 - 80° C and pressure range of 0. 6 - 0. 7 atm.

Detailed description of the invention

The Tea shoots used in the present invention for manufacturing of green tea is obtained from *Camellia sinensis* and *Camellia assamica* varieties. The term tea shoots used in the invention refers to upper two to three leaves and an apical bud including stem portion.

The process to manufacture natural flavored green tea comprises of following steps:

a) Firstly, fresh plucked tea shoots were treated (allowed to wither) under controlled temperature and pressure so as remove excess moisture from leaves by arresting the enzyme activity. The temperature was strictly kept in between the range of 60 - 70° C, while atmospheric pressure was kept at 0.6 - 0.7 atm to get natural flavored tea.

b) Rolled the treated withered tea shoots (step-a) in controlled conditions for specific time period ranging from10-20 minutes, and preferred time is 10-15 minutes so as to get maximum flavors without loosing natural green color of the tea.

c) Finally the rolled tea shoots were dried under controlled conditions of temperature and atmospheric pressure. The drying temperature was kept in range of 60 – 80° C, while atmospheric pressure was kept at 0.6 - 0.7 atm.

d) The dried made tea gives a very clear and natural light green color infusion which is full of natural flavors and aroma.

e) A chamber or cabinet with facility for controlling temperature and atmospheric pressure could be used to perform above mentioned process of green tea manufacture.

Examples

The green tea was manufactured according to the present invention as presented by following examples:

Example 1

This example demonstrates the production of flavored green tea.

For manufacturing of natural flavored green tea 500gm of fresh tea shoots (two to three leaves and a bud) were collected. The tea shoots were put in a controlled chamber adjusted at specific temperature between 60° C and atmospheric pressure was maintained continuously between 0.6 atm. The shoots were allowed to wither for 2 hrs by arresting enzyme activity. The withered shoots under controlled temerature and atmospheric pressure were immediately rolled for as short as possible (10-15 minutes). The rolled tea immediately put in to same drier for drying at controlled temperature (80° C) and controlled atmospheric pressure (0.7atm.). In this way a natural flavored novel green tea was produced which when brewed in hot water (80-90°C) gives a clear, natural green colored and flavored infusion.

Example 2

This example also demonstrates the production of flavored green tea.

1kg of fresh tea shoots (two leaves and a bud) were taken for manufacturing of green tea with enhanced aroma and flavors. The tea shoots were first put in to a chamber whose initial temperature was adjusted between 70° C and atmospheric pressure was maintained continuously between 0.7 atm. The shoots were allowed to wither for 3 hrs by arresting enzyme activity. The shoots were taken out after withering and then rolled immediately for very small duration (10-15 minutes). The rolled tea was immediately put in to same chamber for drying again at controlled temperature (70° C) and controlled atmospheric pressure (0.6 atm.). In this way a natural flavored novel green tea was produced which when brewed in hot water (80-90°C) gives a clear, natural green colored and flavored infusion.

Example 3

This example demonstrates the analysis of major non-volatile constituents of green teas by HPLC analysis

The HPLC analysis of the novel green tea (GT-A) was performed for the estimation of major phytoconstituents present in it. The inventors have collected three differiantly manufactured green tea samples.

1. Green tea leaves processed in oven (GT -B)
2. Green tea leaves processed by steaming (GT-C)
3. Green tea leaves processed by pan roasting (GT-D)

All these four types of green teas (GT-A, GT-B, GT-C and GT-D) were compared for the presence of major phytoconstituents viz. catechins, caffeine and theanine in it using RP-HPLC.

Extraction of green teas for sample preparation

Weighing accurately about 1 gm of the entire four green tea samples were taken. They were ground to powder form using mortar and pestle followed by solvent extraction. The extraction was performed by maceration using 70% acetone as solvent. The three subsequent extractions were performed using 20, 20 and 10ml of extrcation solvents. The extracts were filtered and pooled. From the pooled filtrate the solvent was removed with the help of Buchi Rota vapor and the remaining aqueous fraction was again made up to 50ml using distilled water. In this way all the four teas extracts were prepared.

These above obtained extracts of all the four types of green teas were filtered by 0.45 micron nylon membrane filter prior to HPLC analysis.

RP-HPLC analysis

All the four green teas GT-A, GT-B, GT-C and GT-D were analysed by using Waters HPLC system. The acetonitrile and water with 0.01% TFA were used as solvents. The HPLC conditions were as starting from 10% A

at 0 min. the solvent gradient was increased slowly at rate of 5ml. It was increased from 10% to 15% at 3 min. and to 20% at 5 min, and then increased to 25% and 30% at 8 and 10 min. respectively. The gradient was maintained at 30% A and 70% B for two minutes and then it was again decreased to 20% of A at 16min. to 15% A at 18 min. and finally the gradient was again brought to 10% of solvent A up to 20 min. The injection volume was kept at 10µl.

The HPLC chromatograms of all the four teas viz. GT-A (figure 1), GT-B (figure 2), GT-C (figure 3) and GT-D (figure 4). The results of HPLC analysis were calculated using linear regression equations (y =mx +c); [where y=peak area, x=concentration of standard (micrograms per millilitre) and m and c are the constants] plotted for each individual standard.The data recording amount of theanine, caffeine and catechins different sample of green teas (GT-A, GT-B, GT-C and GT-D) were tabulated in table 1. It is evidenced from the HPLC data that among all the four types of green teas the green manufactured following present invention, does contains comparable amount of catechins and caffeine but relatively higher content of theanine in comparision to other tea samples (Table 1).

Table 1

	GT-A mg/gm	GT-B mg/gm	GT-C mg/gm	GT-D mg/gm
Theanine	10	4.16	9.49	4.16
GC	2.1	2.02	1.88	1.15
EGC	18.56	19.34	22.3	15.6
Catechin	0.46	0.289	0.224	0.06
Caffeine	17.3	14.05	17.53	16.85
EC	4.76	3.74	5.46	4.02
EGCG	20.59	19.5	19.8	16.8
ECG	10.38	9.2	9.59	7.83

Table 1 shows the amount of major constituents of tea in different samples of green tea (GT-A, GT-B, GT-C and GT-D).

Example 4

This example demonstrates the analysis of main volatile constituents of green teas by GC and GC-MS analysis

The GC and GC-MS analysis of all the four green teas viz. GT-A, GT-B, GT-C and GT-D were performed by the inventors for the determination of major volatile constituents present in them. Further all these four types of

green teas (GT-A, GT-B, GT-C and GT-D) were compared for the presence of major volatile constituents.

Extraction of volatile constituents from green teas by simultaneous distillation extraction (SDE)

For the extraction of volatile constituents from green teas inventors have performed simultaneous distillation extraction using modified Likens and Nickerson apparatus (Rawat et al., 2007) with an extended condenser and an air vent connected to a vacuum pump. The coolant of the condenser was maintained at 5 °C with the help of a circulatory water bath (Plasto Crafts make, model No. LTB 20, Mumbai, India). Before SDE extraction, 1.0 mL ethyl caproate solution (5 µL ethyl caproate of in 100 mL of HPLC-grade dichloromethane) was added to the tea as an internal standard. One hundred g of orthodox black tea was placed in a round-bottom flask of 1 L capacity along with 1 g of sucrose dissolved in 700 ml of distilled water. 20 ml of HPLC-grade dichloromethane was added in solvent flask. The heating mantles (Perfit India, 200 watts, 500 ml capacity) of the solvent and sample flasks were maintained at 60–70 and 75–80 °C, respectively. The solution was dried overnight using anhydrous sodium sulfate (Merck, Mumbai India). Volatile compounds extracted by SDE as mentioned above were pooled concentrated finally concentrated to 5 uL using nitrogen flushing and injected into GC and GC-MS for analysis.

The GC profiles of volatile constituents present in different samples of green teas viz. GT-A (figure 5), GT-B (figure 6), GT-C (figure 7) and GT-D (figure 8) and the data was given in table 2. The GC profiles of novel green tea (GT-A) clearly shows dominace of various volatile compounds in comparison to other green samples. The dominance of O-xylene, 1-Octanol, 3, 5-Octadien-2-one, α-Ionone and 6, 10, 14, Trimethyl-2-Pentadecone like volatiles in GT-A by GC/MS analysis (Table 2) have shown that this green tea has more aroma and flavor characters. These results cleary showed that using this process methodolgy for preparing green tea, one can enhance the overall green tea quality without extraneous aduteration with additional advantage of energy efficiency.

Table 2

Compounds	RRI	GT A	GT B	GT C	GT D
o-Xylene	-	1.32	0.37	0.92	0.87
1-Ethyl pyrrole	-	0.87	2.38	5.3	0.78
cis-Pent-2-enol	-	2.32	2.01	2.35	5.88
2,4-Pentadienal	807	1.35	1.29	3.9	2.85
Octanal	1022	0.85	1.77	0.65	1.35
Benzeneacetaldehyde	1045	4.44	1.21	5.84	0.46
Limonene	1046	0.85	1.87	1	2.5
Acetophenone	1085	0.83	0.17	0.25	0.23
1-Octanol	1092	1.66	0.31	0.13	0.55
3,5-Octadien-2-one	1114	1.16	0.92	0.81	0.95
3-Ethyl-2,5-dimethylpyrazine	1120	0.25	0.21	0.08	0.64
Nonanal	1127	0.82	0.63	0.13	0.17
2-Hexenal	1226	2.3	0.23	0.35	nd
1-Pentanol	1257	0.25	0.35	0.43	0.16
3-Hexenol	1386	3.13	2.64	3.64	1.64
2-Ethyl-5-methyl-pyrazine	1393	0.15	0.25	0.73	0.1
cis-Linalool oxide	1440	5.02	6.35	6.65	5.4

trans-Linalool oxide	1468	3.25	2.61	3.15	5.35
Neryl acetone	1478	1.32	1.72	0.08	0.25
2, 4-Heptadienal	1497	0.65	0.13	0.35	0.25
2-Acetyl furan	1500	1.23	1.31	1.32	2.21
Linalool L	1551	10.41	10.28	10.72	9.35
Dihydroactinidiolide	1557	0.91	0.67	0.46	0.62
3,4-Dimethylcyclohexanol	1594	1.16	0.1	0.76	0.2
Hexadecane	1600	1.88	1.35	0.64	0.33
1-Ethyl-2-formyl pyrrole	1605	1.9	0.24	1.06	1.31
Hotrienol	1611	1.55	0.88	0.82	1.61
β-Cyclocitral	1619	0.63	0.85	0.74	0.1
Safranal	1635	2.79	2.25	1.35	1.6
trans-2-Hexenyl caproate	1666	1.72	1.25	1.37	1.55
α-Terpineol	1694	1.15	0.24	0.24	0.4
α-Cadinol	1690	1.07	0.29	0.35	0.64
Epoxylinalool	1762	3.56	3.23	3.82	4.12
Methyl salicylate	1769	0.88	0.35	0.08	0.12
Nerol	1800	0.76	0.44	0.05	0.31
Nonadecene	1826	0.35	3.63	5.51	2.81
Eicosanol	1838	0.45	2.11	2.35	3.64
α-Ionone	1840	1.85	0.62	1.15	0.17
trans-Geraniol	1850	15.36	14.35	16.32	13.25
Phenylethyl alcohol	1911	2.65	2.6	1.72	1.74
cis-Jasmone	1917	1.79	1.51	1.82	1.64
β-Ionone	1933	1.51	1.67	0.96	1.46
Isopropyl myristate	2040	0.74	0.64	0.35	0.15
Nerolidol	2049	3.5	2.9	4.74	3.65
Cubenol	2067	0.64	0.56	0.15	0.55
6,10,14-Trimethyl-2-Pentadecanone	2127	5.09	0.44	1.13	0.54
Docosane	2200	0.35	1.93	1.16	0.17
Phytol	2623	0.25	0.47	0.13	0.19
Composition (%)		98.92	84.58	98.01	84.81

Table 2 shows major volatile constituents in different samples of green tea (GT-A, GT-B, GT-C and GT-D).

Advantages

The present invention has following advantages.

1.) It provides an energy efficient process for manufacturing of enhanced flavored green tea.

2.) The present invention discloses a process for manufacturing of high quality flavored green tea.

3.) The invention disclosed standardization of condition during various steps of tea manufacturing viz. withering, rolling and drying.

4.) The green tea manufactured following present invention is rich in flavor and aroma characteristics.

5.) The present invention leads to the manufacturing of green tea with enhanced organoleptic properties.

6.) A high quality green tea with enhanced aroma and flavors and without any kind of extraneous adulteration.

Claims

1) An energy efficient process for manufacturing of high quality green tea with enhanced flavors which comprises the steps of:
   a) withering freshly plucked tea shoots at controlled temperature and pressure for 2-3 hours , wherein temperature is in the range of 60 - 70° C and atmospheric pressure is in the range of 0.6 - 0.7 atm,
   b) rolling the treated tea (controlled withered) under controlled condition for specific time period ranging 10 -15 minutes
   c) drying of rolled tea at controlled temperature and pressure, at temperature range of 60 - 80° C and pressure range of 0. 6 - 0. 7 atm to get the desired green tea.

2) An energy efficient process as claimed in claim 1wherein a chamber or cabinet with facility for controlling temperature and atmospheric pressure is used to maintain desired temperature and pressure of steps (a) and (c) to get desired green tea .

3) An energy efficient process as claimed in claim 1wherein enzyme activity of fresh tea shoots being arrested during withering for the manufacturing of flavored green tea under controlled temperature at the range of 60 - 70° C and atmospheric pressure is in the range of 0.6 - 0.7 atm.

4) An energy efficient process as claimed in claim 1wherein tea shoots used comprises upper two to three leaves and an apical bud including stem portion.

5) An energy efficient process as claimed in claim 1 wherein tea shoots used is obtained from *Camellia sinensis* and *Camellia assamica* varieties

6) An energy efficient process as claimed in claim 1 wherein green tea obtained at step (C) is free from unpleasant roasted flavor as compared to pan roasted and heat treated green teas.

7) An energy efficient process as claimed in claim 1 wherein the made tea obtained from green tea of step (c), by being brewing the said green tea in hot water (80-90°C) , gives a very clear and natural light green color infusion which is full of natural flavors and aroma.

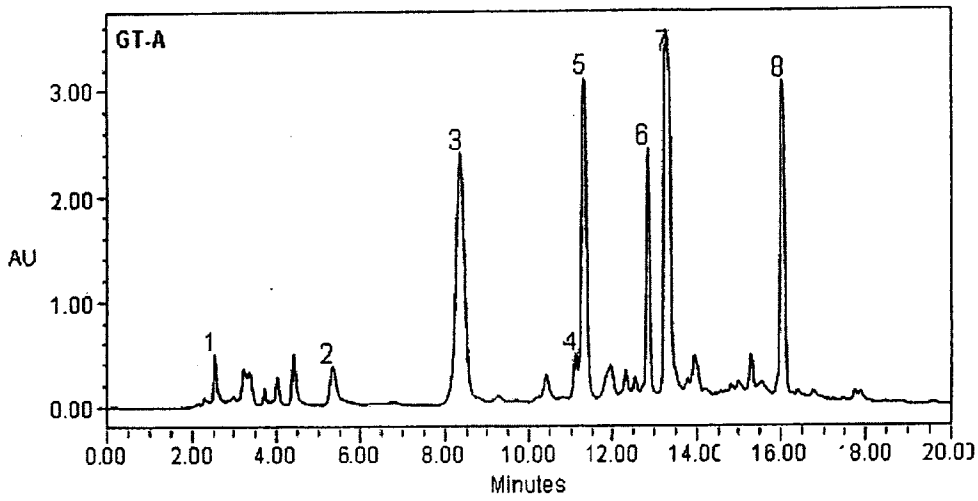
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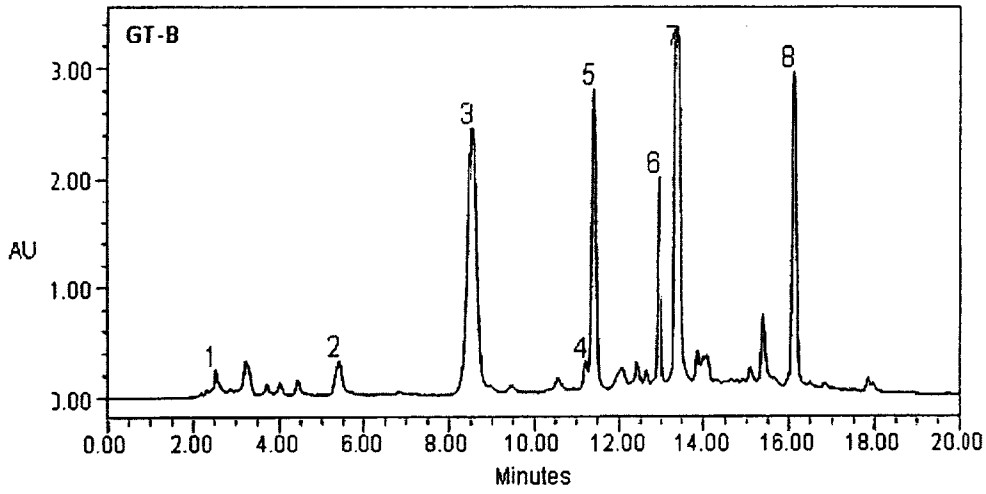
Figure 1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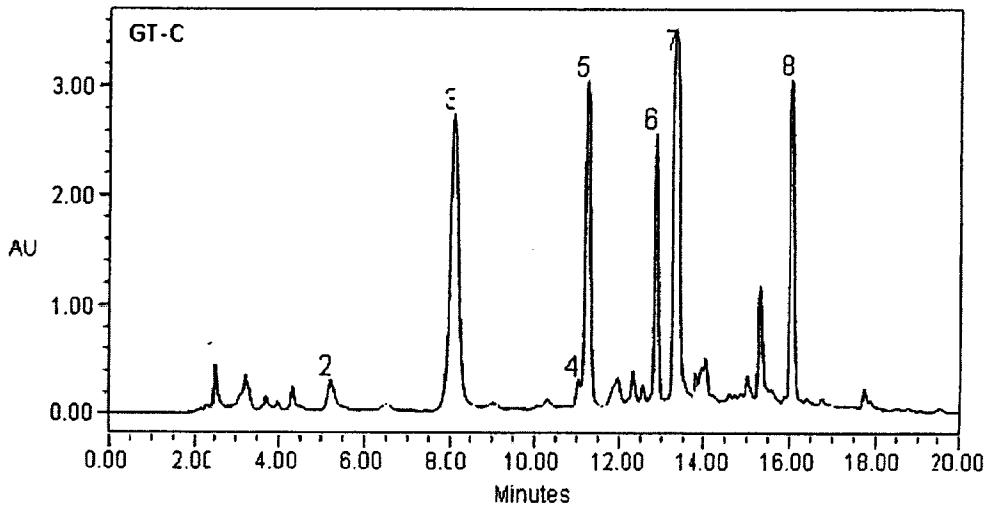
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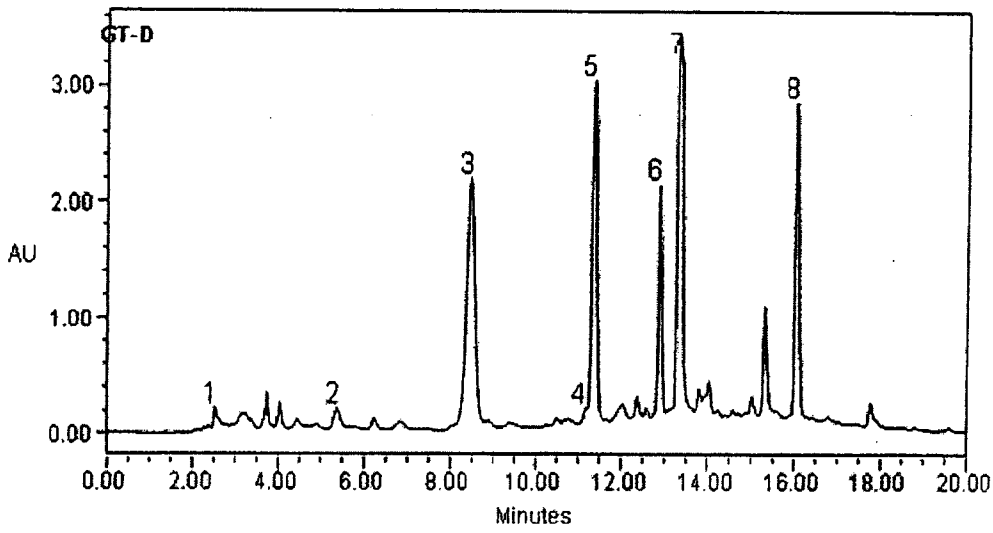
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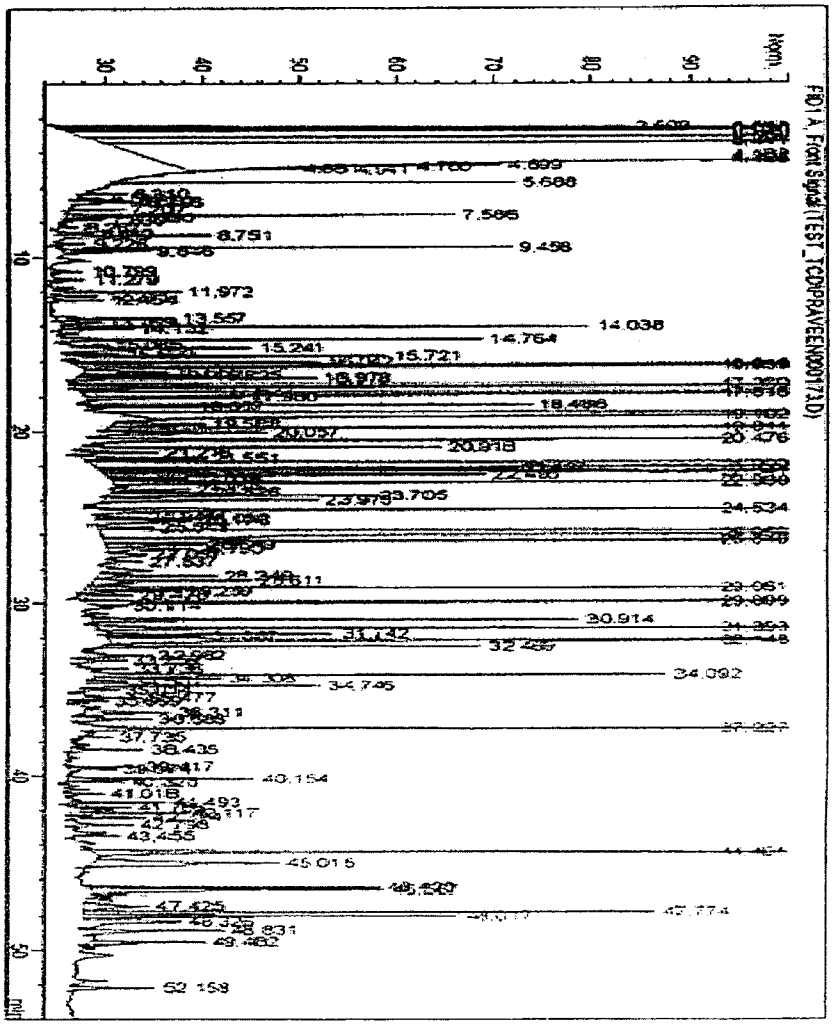
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15.721
18.486
20.476
20.918
23.705
24.534
30.914
32.489
34.092
34.745
36.311
37.735
38.435
40.154
41.018
43.455
45.015
47.425
49.462
52.159

Figure 5

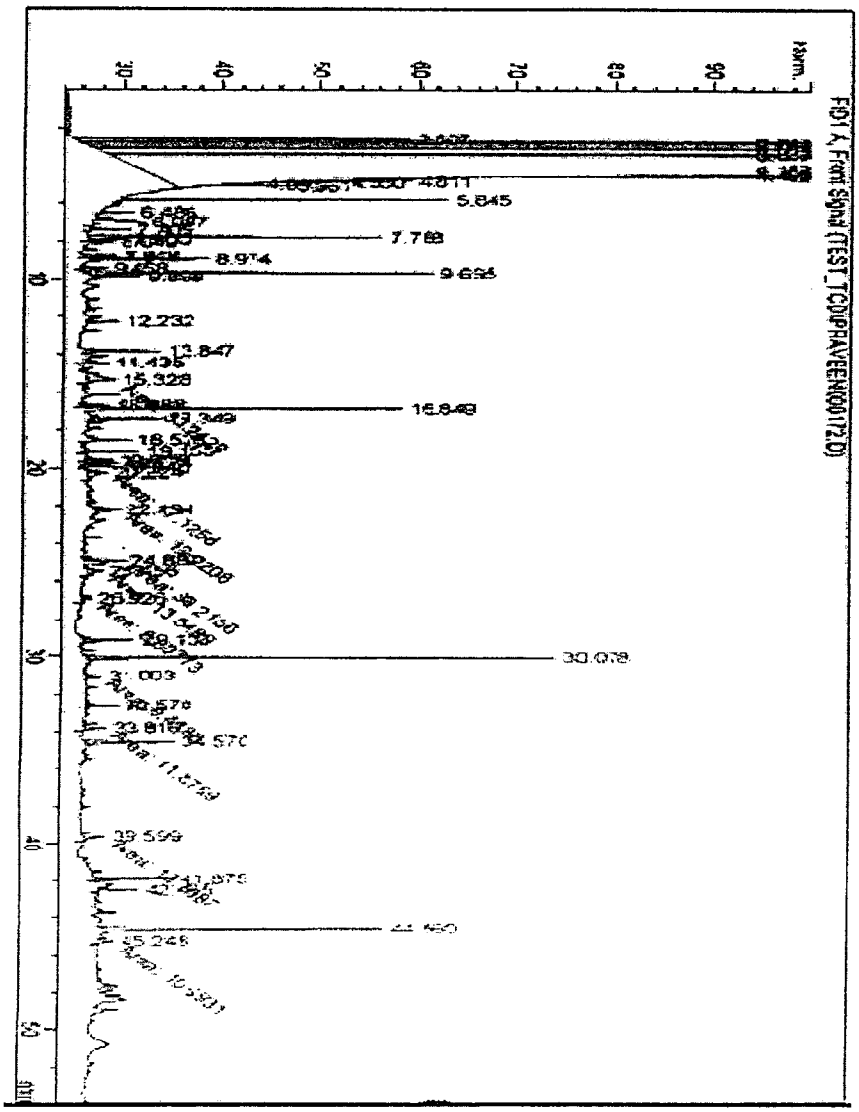
FB1A_Front Signal (TEST_TCM\PRAVEEN\000\12.D)
5.845
7.788
8.974
9.695
12.232
16.849
30.078
39.599
41.450

Figure 6

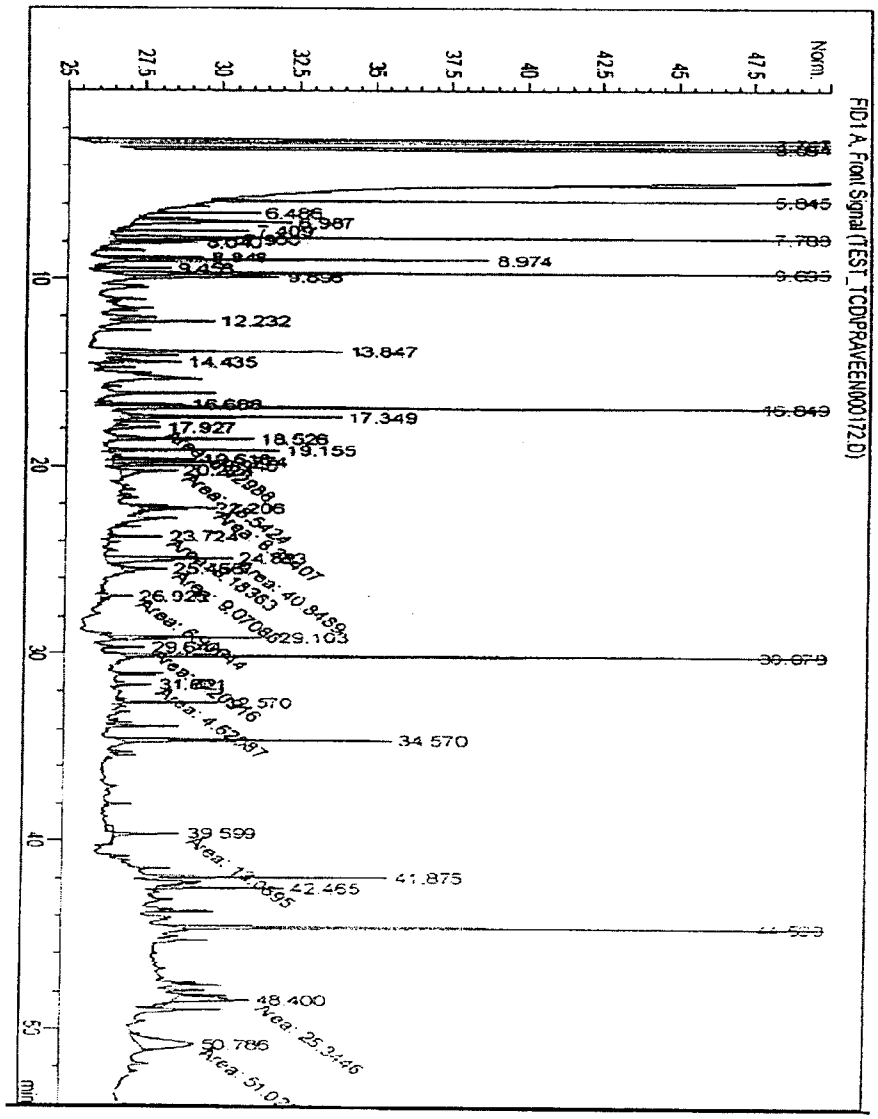
25
27.5
30
32.5
35
37.5
40
42.5
45
47.5
Norm.
FID1 A, Front Signal (TEST_TCD\PRAVEEN\003172.D)
6.486
8.974
12.232
13.847
14.435
17.349
18.526
19.155
23.724
34.570
39.599
41.875
42.465
48.400
50.786
min

Figure 7

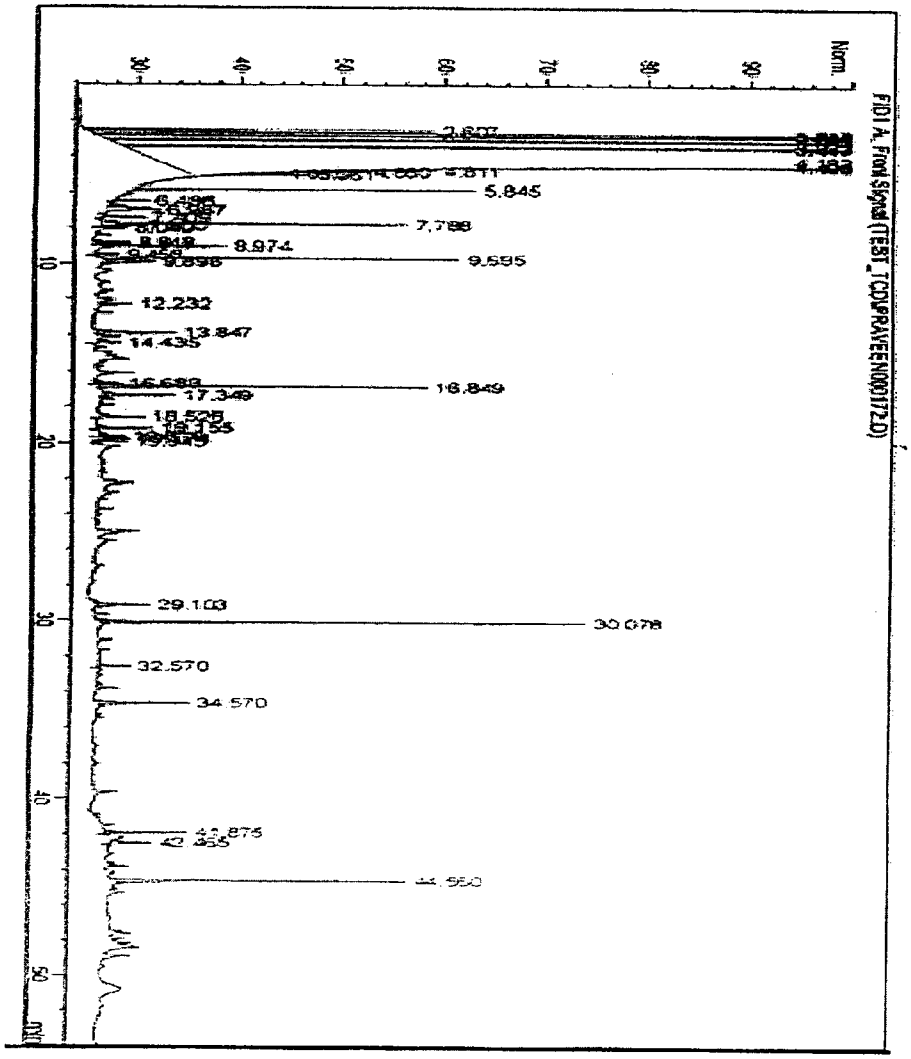
Norm.
FID1 A, Front Signal (TEST_TCD\PRAVEEN\0001T2.D)
2.607
5.845
7.788
8.974
9.595
12.232
13.847
14.435
16.849
17.349
29.103
30.078
32.570
34.570
42.875
43.555
44.950

Figure 8

INTERNATIONAL SEARCH REPORT

International application No
PCT/IN2015/000078

A. CLASSIFICATION OF SUBJECT MATTER
INV. A23F3/06
ADD.

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)
A23F

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)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2000 004788 A (YAGI SHUNICHI) 11 January 2000 (2000-01-11) claims 1-4; examples 1-3	1-7
X	JP 2002 034455 A (SATOEN KK) 5 February 2002 (2002-02-05) paragraph [0011]; claims 1-5	1-7
X	JP 2012 050412 A (OKIYU INC) 15 March 2012 (2012-03-15) paragraph [0009]; claims 1-11	1-7

☐ Further documents are listed in the continuation of Box C.

☒ 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

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"T" 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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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 June 2015	24/06/2015

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/IN2015/000078

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 2000004788 A	11-01-2000	NONE	
JP 2002034455 A	05-02-2002	NONE	
JP 2012050412 A	15-03-2012	JP 5711922 B2	07-05-2015
		JP 2012050412 A	15-03-2012