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Ultrasonic Assisted Extraction of Jatropha Seed Oil and Analysis of Oil Component by HPLC

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Ultrasonic extraction technique was applied for the extraction of oil from Jatropha seeds.The optimum conditions for the lab scale ultrasonic extraction were obtained at 30 °C, ultrasonic extraction time of 4 min, ratio of liquid to solid of 14:1(mL/g) and ligarine (90-120 °C) as a solvent. Under the optical conditions, the yield of oil was 42.52%. The main components of jatropha seed oil were analyzed by the HPLC method.

Keywords: Jatropha seed, Extraction, Oil, Ultrasonic, Analysis.

INTRODUCTION

With the decreasing of natural resources, the extraction of useful substances from natural materials increasingly a cause for concern, especially in the traditional chinese medicine extraction, petroleum chemical industry. Traditional extraction technology is not only time-consuming but also the extraction rate is not high, wasting large amounts of solvent[1]. Compared to traditional extraction techniques, in resent years, the development of ultrasound-assisted extraction technology has been greatly improved and it can significantly improve the extraction rate[2]. This paper discusses the the ultrasound-assisted extraction efficiency under different conditions in the scope of the laboratory and provides a reference for future applications of ultrasound in the industry and the main component analysis by the HPLC method.

EXPERIMENTAL

Rotary evaporators, ultrasonic generator, vacuum pump, water bath, funnel, rubber tube, condenser pipe and other glassware instruments, Agilent 1200 HPLC system, Agilent C18 Column (3.5 μm, 100 mm × 4.6 mm i.d.).

Jatropha seeds were purchased from Guizhou province of China. The sample was ground in a blender to produce a fine powder. Ligarine (30-60 °C), ligarine (90-120 °C), diethyl ether, dehydrated alcohol, ethyl acetate and *n*-hexane were purchased from the Nan Jing Jian Chen Biotechnology Lt. Cod. Methanol (HPLC Grade) was purchased from Romil Ltd.

Choice of solvents: To study the effect of solvents on the yield of the extract, six portions of Jatropha seeds powder were respectively placed in six 250 mL round bottom flasks, which portion weighted 10 g. Then 80 mL solvents (diethyl ether, dehydrated alcohol, ligarine (30-60 °C), ligarine (90-120 °C), ethyl acetate, *n*-hexane) were respectively added into the flasks. The extracts were in the flasks in a ultrasonic generator for extraction (20 °C, 2 min) and vacuum filtered to remove solvents. Evaporation of the organic solvent under reduced pressure gave the Jatropha seeds oil, which was weighed to give the yield of the extract.

$$\text{Yield of the extract (\%)} = \frac{\text{Jatropha seeds oil (g)}}{\text{Jatropha seeds powder (g)}} \times 100$$

As shown in Fig.1, maximum yield of the extract was achieved when ligarine (90-120 °C) was used and minimum yield of the extract was achieved when dehydrated alcohol was used. Therefore, ligarine (90-120 °C) was used as the solvent for Jatropha seeds oil production in the experiment.

Effect of temperature on the yield of extract: Extraction temperature was set at 20, 30, 40, 50, 60 °C, respectively. Ligarine (90-120 °C) was chosen as solvent. Five portions of Jatropha seeds powder were respectively placed in five 250 mL round bottom flasks, which portion weighted 10 g. Then 80 mL ligarine (90-120 °C) were respectively added into the flasks. The extracts were in the flasks in an ultrasonic generator for extraction (5 min) and vacuum filtered to remove solvents.

Effect of ultrasonic time on the yield of extract: Ultrasonic time was set at 2, 4, 6, 8, 10 min, respectively. Ligarine (90-120 °C) was chosen as solvent for extraction (20 °C) and other conditions are the same to those described in effect of temperature on the yield of the extract.

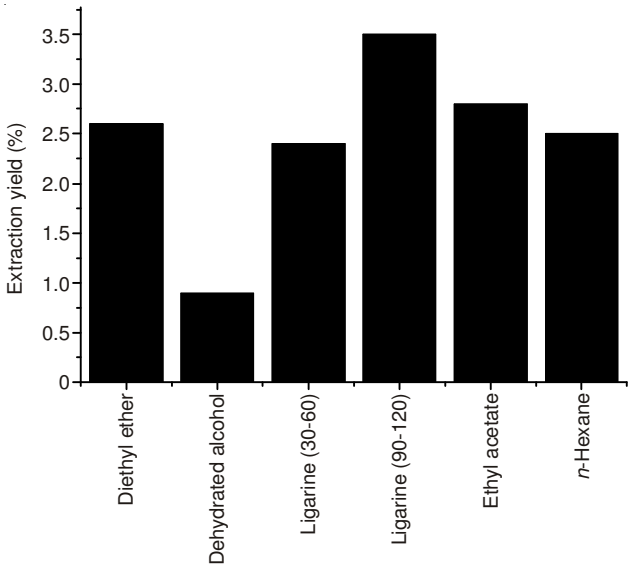

Fig. 1. Effect of solvent on amount of extracted oil

Effect of ratio of liquid to solid (v/m) on the yield of the extract: Ratio of liquid to solid (mL/g) was set at 6:1, 8:1, 10:1, 12:1, 14:1, respectively. Ligarine (90-120 °C) was chosen as solvent for extraction (20 °C) and other conditions are the same to those described in effect of temperature on the yield of the extract.

Optimum of the experimental conditions: On the basis of single-factor test, an attempt was made to optimize three parameters, which are confirmed to significantly affect the yield of the extract, as extraction temperature, ultrasonic time, ratio of liquid/solid to obtain good yield of Jatropha seeds oil by orthogonal test. Each of these parameters was varied at three levels: temperature at 30, 40 and 50 °C, ultrasonic time at 2, 4 and 6 min, ratio of liquid/solid at 10/1, 12/1 and 14/1.

Chromatographic conditions: The chromatographic elution was accomplished isocratically with methanol-water (99:1 v/v) at a flow rate of 1 mL/min. The temperature was maintained at a room temperature and the injection volume was 20 µL. DAD detection was 205 nm.

RESULTS AND DISCUSSION

Assay of effect of extraction temperature on the yield of the extract: To investigate effect of extraction temperature on the yield of Jatropha seeds oil, a study was conducted by employing different extraction temperatures in the range of 30 to 50 °C (Fig. 2). As can be seen, the yield of oil increased with increasing temperature and reached a peak value at 40 °C. The percent oil yield inversely decreased after this optimum temperature value. Therefore, 40 °C was chosen as optimum extraction temperature.

Assay of effect of extraction ultrasonic time on the yield of the extract: Fig. 3 shows the effect of extraction time on the yield of the extract. Under the given conditions, there was marked correlation between the extraction time and the extraction yield of oil. The yield of the extract was found to increase from 24.325 to 31.974 % when the extraction time increased from 2 to 6 min. The yield of the extract no longer increased with further increase in extraction time, suggesting

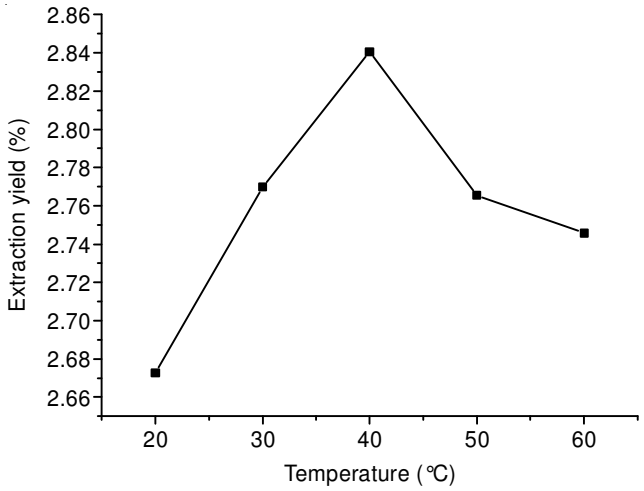

Fig. 2. Effect of temperature on the yield of the extract

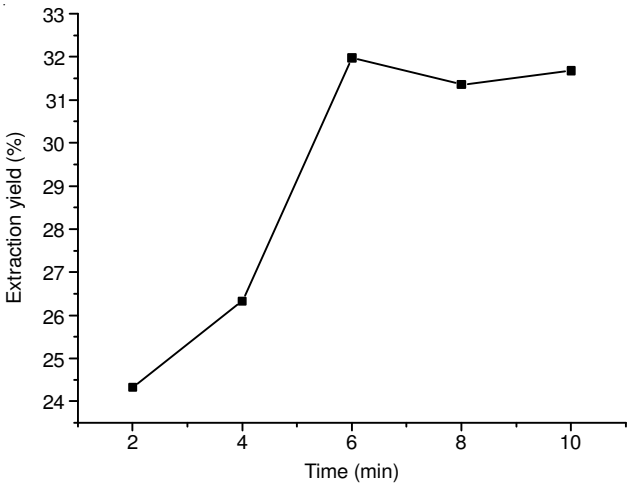

Fig. 3. Effect of ultrasonic time on the yield of the extract

that when Jatropha seeds powder was extracted for 6 min, remnant oil contained in powder particles had been dropped to a minimum value. According to the obtained results, extraction time was chosen between 2 and 6 min for later orthogonal optimum tests.

Assay of effect of ratio of solvent to solid on the yield of the extract: In order to determine the optimal ratio of solvent to solid, the effects of ratio of solvent to solid on the extraction yield was examined as shown Fig. 4. It can be observed that the extraction rate gradually increased with increasing ratio of solvent to solid and reach the maximal value as the ratio of solvent to solid was 10:1 (mL/g) under the given condition.

Optimum of extraction technology of Jatropha seeds oil: Based on the analytical data obtained in Table-1, the optimal extraction condition was determined as A3B1C1 when all levels of the three factors were considered. Therefore, the optimal parameters combinations are as following: ratio of liquid to solid of 14:1(mL/g), temperature 30 °C, ultrasonic time 4 min. Further variance analysis shows that the decreasing order of effect of the three factors on extraction yield of Jatropha seeds oil were C > A > B.

There were there parallel experiments to be done under the optimal extraction condition. The mean of three parallel experiments is 42.52 %.

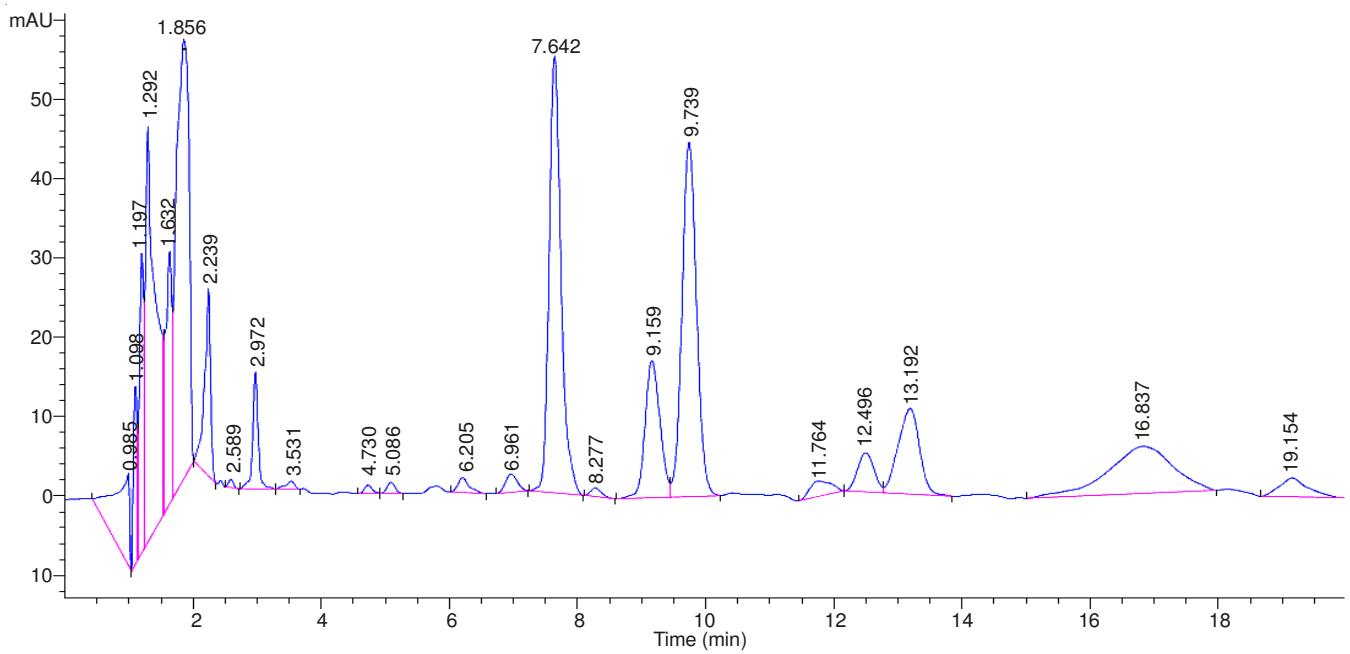

Fig. 5 UV chromatograms of Jatropha seed oil

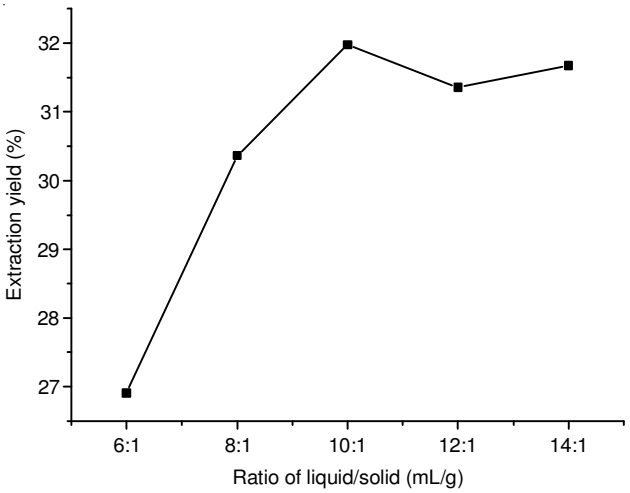

Fig. 4. Effect of ratio of liquid/solid on the yield of the extract

TABLE-1
OPTIMUM OF EXTRACTION PARAMETERS
OF JATROPHA SEEDS OIL

No.	Factors			Extraction rate (%)
	A	B	C	
1	10 : 1	4	30	35.057
2	12 : 1	4	40	32.579
3	14 : 1	4	50	36.013
4	14 : 1	6	30	32.600
5	10 : 1	6	40	36.103
6	12 : 1	6	50	34.753
7	12 : 1	8	30	40.240
8	14 : 1	8	40	34.245
9	10 : 1	8	50	36.927
K1	34.55	35.97	34.69	
K2	34.49	34.31	34.04	
K3	37.14	35.90	28.518	
R	0.265	0.166	0.341	

Assay of main components: As shown in Fig. 5, there were many components in the Jatropha seed oil. The components which retention time were smaller than 3 min were free fatty acid. Which retention time was almost 7 min were plant sterols by the β-sitosterol standard. The components which retention time were more than 10 min were triglyceride.

Conclusion

The decreasing order of effect of the three factors on extraction yield of Jatropha seeds oil were temperature > ratio of liquid/solid > ultrasonic time. The optimum conditions for the lab scale extraction were obtained ligarine (90-120 °C) as solvent, extraction temperature 30 °C, ultrasonic time 4 min and ratio of liquid/solid of 14:1 (mL/g). The extraction yield of Jatropha seeds oil was 42.52 %. The main components of jatropha seed oil were free fatty acid, plant sterols and triglyceride by the HPLC method.

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