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Ultrasonic Assisted Extraction- Gas Chromatography-Mass Spectrometry Analysis of Chemical Components of Dandelion Root

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The components of dandelion root were extracted by ultrasonic-assisted extraction method and its chemical compositions were analyzed by GC-MS. Based on the single experiment, the extraction yield of components in dandelion root as the response value, the best condition of chemical components extraction was optimized by orthogonal array design and analysis. At last, the results showed that ultrasonic extraction time had the greatest impact on the extraction yield of chemical components, followed by the volume ratio of ethanol to ether, material-liquid ratio affected least. The optimum extraction conditions were the volume ratio of ethanol to ether 2:5, ultrasonic time 15 min, and material-liquid ratio 1:35 (g:mL), Under this condition, the average value of the experiment turned out to be 7.72%; Totally 17 compounds were identified by GC-MS, accounting for 89.97% of the total separated compounds. The major compounds identified were Germanicus(34.84%); Olean-18- en-28-olc acid, 3-oxo-, methyl ester(16.21%); 2H-1-Benzopyran-2-one, 6-acetyl-7 -(acetyloxy)-4-methyl-(14.14%); urs-20-en-3-ol(3β,18α,19α)(11.49%); 6S-2,3,8,8- Tetramethyltricyclo [5,2,2,0](1,6)]undec-2-ene(2.15%); Genistein(1.51%); α-Amyrin (1.48%); urs-20-en -3-ol (3β,18α,19α)(1.38%), ect.

1. Introduction

Dandelion is one of perennial herb belonging to composite, dandelion has about more than 100 varieties in Chinese, producing almost all over the country. Traditional Chinese medicine believes that dandelion bitter taste, sweet, cold, with heat clearing and detoxicating, detumescence, diuresis effect, use for swollen poison, mastitis, eye pain, sore throat, appendicitis and jaundice, etc (You et al., 2010). Modern clinical research shows that dandelion can treat a variety of infectious diseases (Trojanova et al., 2004; Jennifer, 2015; Munkhtugs et al., 2013), dandelion herb showed broad-spectrum antibiotic effect on certain diseases (Xu et al., 2000; Wang, 2014;). In addition, dandelion can also be used as food, health care products, cosmetics, and has good economic benefit. Many studies have indicated that the main chemical ingredients of dandelion are triterpenoids, flavonoids, coumarins, phenolic acids, fatty acids, sesquiterpene lactones and other substances, these are also the major pharmacologically active ingredient of dandelion (Rblert et al., 2010; Tomoyoshi et al., 1994; Klaudia et al., 2010; ZielinÂ and Kisiel, 2000;).

The common method of extracting chemical constituents from the plants were steam distillation, supercritical CO2 fluid extraction (Krunoslav et al., 2015; Juliana et al., 2016;), microwave extraction method (Avelina et al., 2016;), ultrasonic solvent extraction (Xu et al., 2015;), etc. Although Steam distillation method have simple equipment, low energy consumption, but low extraction rate and the distillation time is too long; Supercritical CO2 extraction method using CO2 as solvent, no solvent residue, but not suitable for extraction of nonpolar substances and high operating cost. Ultrasonic extraction method can accelerate the dissolution rate of active ingredients in plant. The secondary effect of ultrasonic such as mechanical vibration, emulsification, diffusion, smash and chemical effect also can accelerate the release of active ingredients and fully mixed with the solvent, and this method has good safety, simple operation, convenient maintenance, no need of high temperature, high extraction efficiency.

We chose ultrasonic assisted extraction solvent extracted the chemical composition of dandelion root and the extraction process was optimized. Chemical constituents of dandelion root were separated and identified by

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gas chromatography-mass spectrometry, in order to provide theoretical reference for research in medicine and pharmacology of the dandelion.

2. Material and Methods

2.1 Materials and instruments

Materials: Fresh dandelion root; ether (A.R); ethanol (95%; A.R).
Main instruments: gas chromatography mass spectrometry instrument of the Thermo Scientific co., LTD (PolarisQ ITQ1100); Electronic analytical balance of The United States mettler Toledo instrument (Shanghai) co., LTD(FA1104N); Circulating water vacuum pump (SHB-III); Ultrasonic cleaner(KQ100VDB); Electric air blast drying box (DHG-90 70); Universal mill of Chinese herbal medicine

2.2 Extraction of chemical constituents from dandelion root

The fresh dandelion root is dried at 40℃, then cut and broken, with a net sieve filter, take 1 grams of dandelion powder(md) in a solvent extraction bottle(m1) and then ultrasonic extraction, filtration, the filtrate was dried (m2) with anhydrous Na2SO4 and then to be detected. Calculation formula of yield:

$$\text{Extraction rate}=\frac{m_2-m_1}{m_d}*100\%$$

2.3 Single factor experiment

In this experiment, the effects of the three factors {the volume ratio of ethanol to ether (1:0), (3:1),(1:1),(1:3),(0:1), ultrasonic extraction time (10,20,30,40,50 min) and material -liquid ratio [1:10,1:20,1:30,1:40,1:50(g:mL)]} on the extraction yield of components in dandelion root were respectively investigated.

2.4 Design of orthogonal experiment

On the basis of single factor test, L9 (34) orthogonal table was used to arrange the experiment, and the extraction yield of components in dandelion root was determined, and the orthogonal experiment was carried out to find out the best technological conditions. The factors and levels of orthogonal test are shown in table 1.

Table1: Orthogonal test design of the chemical constituents of dandelion root by ultrasonic assisted extraction

Levels	Factors		
	A the volume ratio of ethanol to ether	B ultrasonic extraction time (min)	C material -liquid ratio (g:ml)
1	2:1	15	1:25
2	2:3	25	1:35
3	2:5	35	1:45

2.5 GC-MS Analysis

GC-MS analysis was conducted on Thermo Fisher Scientific Gas chromatography- mass spectrometry (ITQ1100, and USA). The column used was a TR5-MS column measuring 30×0.25 mm with a film thickness of 0.25 um and composed of 95% dimethyl polyciliate and 5% Phenyl (equiv) polysilphenylene-siloxane. The carrier gas used was helium at a flow rate of 1.5 mL/min. A 1μL sample injection volume was utilized. The inlet temperature was maintained as 280℃. The oven temperature was programmed initially at 40℃, then increased to 180℃ at a rate of 20℃/min. The oven then remained at 180℃ for 15 minutes, then the temperature was programmed to increase to 235℃ at a rate of 6℃/min, then increased to 250℃ at a rate of 10℃/min, with a 1 min period, then the temperature was programmed to increase to 300℃ at a rate of 5℃/min ending with a 1 min period, the total run time was 48.5 min. The MS transfer line was maintained at a temperature of 220℃. The source temperature was maintained at 220℃. The GC-MS was analyzed using electron impact ionization at 70 eV and the data were evaluated using total ion count (TIC) for compound identification and quantification. The spectra of the components were compared with the database of spectra of known components stored in the GC-MS library. Measurement of peak areas and data processing were conducted by using the database of the NIST2005. The names, molecular weights and structure of the components of the test materials were thereby ascertained.

3. Results and Discussion

3.1 Single factor experiment

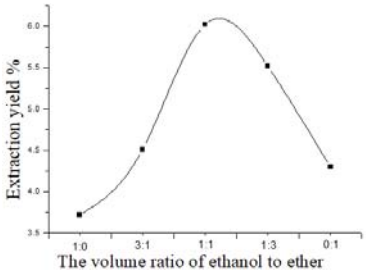

Figure 1: The influence of the volume ratio of ethanol to ether on the yield

The influence of the volume ratio of ethanol to ether on the yield: The effect of different volume ratio on the extraction yield is shown in Figure 1. From Figure 1 it can be seen that the extraction rate was the higher when the mixture of ethanol and ether was used as solvent. This is because the boiling point of the mixed solvent was relatively low, and it was easy to be volatile, which was favorable for the separation of products. Therefore, the mixed solvent was used as the extracting solution and the volume ratio of ethanol to ether was 1: 1.

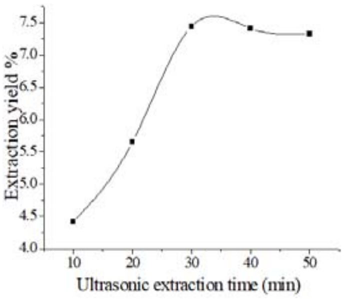

Figure 2: The influence of ultrasonic extraction time on the yield

The influence of ultrasonic extraction time on the yield: The influence of different ultrasonic extraction time on the extraction yield was shown in figure 2, with the ultrasonic treatment time increased from 10 min to 30 min, the extraction rate increased rapidly. When the ultrasonic treatment time was more than 30 min, the extraction rate decreased with the increase of ultrasonic time, this is due to the concentration difference between the inside and outside of the cell, chemical composition was easy to be extracted from the cell in the early ultrasonic extraction. But with the extraction time extended, the concentration difference between the inside and outside of the cell decreases, coupled with the structure of chemical composition has been damaged by the large amount of heat and cavitation -effect generated by ultrasonic wave, which leads to the decrease of extraction yield.

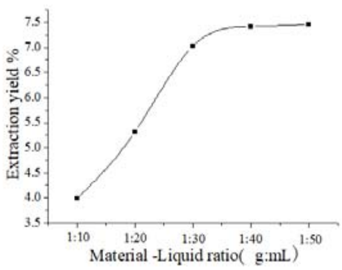

Figure 3: The influence of material -liquid ratio on the yield

The influence of material -liquid ratio on the yield: The influence of different material -liquid ratio on the extraction rate was shown in figure 3. When the ratio of material -liquid was 1:40 (g:mL), the extraction rate of chemical constituents of dandelion was the largest, then the extraction rate changed little with the increase of solvent, the more solvent dosage, the more soluble of chemical composition, but the amount of solvent too much will cause the waste of resources. Therefore, the ratio of material to liquid is more suitable for 1: 40 (g:mL)

Table 2: Results of L9 (33) orthogonal experiment

No .	A the volume ratio of ethanol to ether	B ultrasonic extraction time (min)	C material -liquid ratio (g:ml)	Extraction yield (%)
1	1(2:1)	1(15)	1(1:25)	6.37
2	1	2(25)	2(1:35)	5.09
3	1	3(35)	3(1:45)	5.16
4	2(2:3)	1(15)	2	7.55
5	2(80)	2(25)	3	5.23
6	2(80)	3(35)	1	5.14
7	3(2:5)	1(15)	3	7.32
8	3(100)	2(25)	1	6.05
9	3(100)	3(35)	2	6.35
k1	5.540	7.080	5.853	
k2	5.973	5.457	6.330	
k3	6.573	5.550	5.903	
R	1.033	1.623	0.477	

3.2 Orthogonal experimental design

From table 2, it can be seen that, in the three factors, ultrasonic extraction time had the greatest impact on the extraction rate, then the volume ratio of ethanol to ether, the ratio of material to liquid was the smallest. The influence of each factor on the extraction rate was as follows: ultrasonic extraction time > the volume ratio of ethanol to ether > material-liquid ratio, Therefore, the optimum technological condition was A3B1C2, that is, the volume ratio of ethanol to ether was 2:5, the ultrasonic extraction time was 15 min, the material to liquid ratio was 1:35 (g:mL). Under these conditions, the extraction rate of dandelion root was the largest, and the extraction rate was 7.72%.

3.3 Analysis of chemical constituents from dandelion root

31 peaks (%Area>0.15%) were separated by GC-MS from the dandelion root, the total ion flow diagram of the chemical composition of dandelion root was shown in figure 4, and the identification results are shown in table3. From table 3 it can be seen that 17 compounds were identified, which accounted for 89.97% of the total mass. The main chemical constituents of dandelion root were Germanicol (34.84%); Olean-18-en-28-olc acid, 3-oxo-, methyl ester (16.21%); 2H-1-Benzopyran-2-one, 6-acetyl-7-(acetyloxy) -4-methyl-(14.14%); urs-20-en-3-ol (3β,18α,19α) (11.49%); 6S-2,3,8,8-Tetramethyltricyclo [5,2,2,0](1,6)] undec-2-en e (2.15%); Genistin (1.51%); α- Amyrin (1.48%); urs-20-en -3-ol(3β,18α,19α)(1.38%), ect. The content of vitamin E was also very high in dandelion root, and it reached 1.51% of the total extract mass fraction. Coumarin compounds were also extracted in dandelion root, such as Coumestrol. The research results were consistent with the literature report (Wu and Piao, 2005; Xu et al., 2015; Xu et al., 2000).

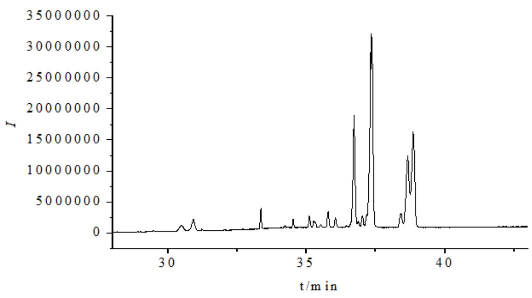

Figure 4: Total ion chromatogram of chemical components of dandelion root

Table 3: Analytical results of chemical constituents in dandelion root

NO.	RT	Area%	Name	Formula	mass
	30.48	1.38	urs-20-en-3-ol(3β,18α,19α)-	C30H50O	426
	30.89	2.15	6S-2,3,8,8-Tetramethyltricyclo [5,2,2,0] (1,6)] undec-2-ene	C15H24	204
	31.21	0.15	2-Bromotetradecane	C14H29Br	276
	33.34	1.51	Genistin	C21H20O10	432
	34.21	0.46	campesterol	C28H48O	400
	34.51	0.69	stigmasterol	C29H48O	412
	35.09	0.97	β-Sitosterol	C29H50O	414
	35.26	0.85	Euparone	C12H10O4	218
	35.51	0.28	Oxazalam	C18H17ClN2O2	328
	35.78	1.48	α-Amyrin	C30H50O	426
	36.04	0.99	α-Amyrin	C30H50O	426
	36.72	14.14	2H-1-Benzopyran-2-one,6-acetyl-7-(acetyloxy)-4-methyl-	C14H12O5	260
	37.01	0.87	t-Eudesmol	C15H26O	222
	37.34	34.84	Germanicol	C30H50O	426
	38.40	1.51	Fenthion sulfoxide	C10H15O4PS2	294
	38.66	11.49	urs-20-en-3-ol(3β,18α,19α)-	C30H50O	426
17	38.85	16.21	Olean-18-en-28-olc acid,3-oxo-, methylester	C31H48O3	468

4. Conclusion

Based on the single factor experiment, the orthogonal experiment was designed, and the chemical constituents of dandelion root were extracted by ultrasonic assisted extraction, the effect of ultrasonic extraction time, the volume ratio of ethanol to ether and the ratio of material to liquid on the yield were investigated, The results showed that the effect of the factors on the yield from large to small is ultrasonic extraction time, the volume ratio of ethanol to ether, the ratio of material to liquid. The optimal extraction conditions for the volume ratio of ethanol to ether was 2:5, ultrasonic extraction time was 15 min, material - liquid ratio was 1:35 (g:mL), under these conditions, the extraction rate of chemical constituents of dandelion root was 7.72%. 31 kinds of components were separated by GC-MS, and 17 compounds were identified, which were confirmed by 89.97% of the total mass fraction. The main chemical constituents of dandelion root were Germanicol (34.84%); Olean-18-en-28-olc acid,3-oxo-, methyl ester (16.21%); 2H-1-Benzopyran- 2-one, 6-acetyl-7 -(acetyloxy)-4-methyl-(14.14%), Taraxasterol (11.49%), Lupeol (2.15%), Coumestrol (1.51%), Vitamin E (1.51%), Hop-22(29)-en-3-one (1.48 %), Taraxerol (1.38%), etc. The main ingredients are triterpene compounds in the dandelion root, and the study of Katrin Schutz (You et al., 2010) and Wu Yan-Ling (ZielinÂ and Kisiel, 2000) proved that triterpene compounds from dandelion root can promote gastrointestinal dynamics in mice.

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