

Optimization of Microwave-Assisted Extraction of Tea Saponin and Its Application on Cleaning of Historic Silks

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Received: 11 March 2013 / Accepted: 7 August 2013 / Published online: 20 August 2013
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Abstract Microwave-assisted extraction (MAE) was utilized to extract tea saponin from oil-tea camellia seed cake. The factors influencing the extraction efficiency were studied, including the effects of microwave power, irradiation duration, temperature, ratio of solvent to material and aqueous ethanol concentration. By systematic orthogonal experiments, the optimal extraction technology was determined. Compared with a conventional extraction method, MAE shows great advantages with the extraction time reduced from 6 h to 4 min, 50 % organic solvent saved and about 14 % extraction yield enhanced. Fourier transform infrared spectroscopy testing and high performance liquid chromatography analysis proved that the extracted resultants were tea saponin with similar compounds as a standard tea saponin. The extracted tea saponin was applied on

the cleaning of historic silks and showed good removal effect on the stains. This work provides useful information for fully use of oil-tea camellia seed cake and new applications of tea saponin at the protection of historic textiles.

Keywords Microwave-assisted extraction · Tea saponin · Oil-tea camellia seed cake · Cleaning

Introduction

Tea saponin is a kind of non-ionic surfactant which has good foaming, emulsifying and decontaminating properties [1, 2]. It plays important roles in daily chemical, food, pharmaceutical and agricultural industries [3, 4]. As a kind of natural source surfactant, it is mild, safe and pollution-free and efficient as detergent. Moreover, it shows quite faint yellow color, which is lighter than most of natural surfactants and could be a candidate to develop detergents for cleaning of old textiles with no or low color influencing and other damages.

The oil tea tree is widely cultivated in Xiangxi, a southwest district in China where the Miao and Tujia minorities live. Every year, plenty of oil-tea camellia seed cakes are produced after the process of oil production and treated as waste residues. However, the residues contain a large amount of tea saponin (about 16 wt%) and other ingredients including water, flavonoid, polysaccharide, etc. [3]. In order to make full use of the wasted oil-tea camellia seed cake, it is important to separate tea saponin from the oil-tea camellia seed cake in an economical, efficient and pollution-free way and to develop its new applications.

Although conventional extraction methods like maceration and stirring extraction, shaking extraction, and heat reflux extraction are widely applied in the separation of tea

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saponin for their availability, large amount of organic solvents were utilized during these extraction processes [5, 6]. These methods were considered as time-consuming, low efficient and expensive [7]. Therefore, people still try to find more available methods for the separation of tea saponin. For instance, Jin Yan and his co-workers [8] applied a two-stage foam fractionation technology to separate tea saponin. Effects of temperature, initial pH, loading liquid volume and superficial airflow rate in the two-stage foam fractionation process were investigated.

Microwave-assisted extraction (MAE) is a process of using microwave energy to heat the solvent in contact with a sample to partition compounds of analytical interest from the sample matrix into the solvent [9, 10]. In MAE, the solvent mixture is heated directly by microwaves, and the direct interaction of microwaves with the free water molecules present in the plant cells, which results in the subsequent rupture of the plant tissue and the release of the active compounds into the organic solvent [11, 12]. Thus MAE has many advantages such as higher efficiency, shorter time, lower cost and environmental friendliness [13, 14].

Since 1985, MAE had been applied in the extraction of compounds from different plants [15]. In the past 20 years, MAE has been frequently used in analytical laboratories [16]. Particularly, the speed of the extraction process using microwave energy and the quality of the extracted compounds make this technique a powerful tool for isolating important pharmaceutical compounds and in other new applications. For instance, the extraction of ginseng saponins [5], volatile compounds [17], and total bioactive saponin fraction from *Gymnema sylvestre* [10] were reported. MAE was also applied in the extraction of tea saponin in China by several researchers [18–20]. They carried out pilot studies on the effects of different factors on the extraction yield. However, in these studies, the authors only depicted the correlative relationship between the yields and some influencing factors. The effects of temperature and the aqueous ethanol concentration had not been discussed. No systematic analysis was being done to make the relationship clear and the highest yield reported was only 12.16 %. The ingredients of resultant extractives were not identified and no product application was reported.

Therefore, we improved MAE in the extraction of tea saponin from waste oil-tea camellia seed cakes. Systematic orthogonal experiments (including five factors) were done after single factor experiments to determine the optimal extraction technology for tea saponin extraction by MAE. The results by the optimal extraction technology were compared with those by a conventional extraction. The contents of the extractives were calibrated. The chemical compositions of the extractives were compared with a

standard tea saponin by Fourier transform infrared spectroscopy. The ingredients of the extractives were tested by high performance liquid chromatography. The extracted tea saponin was applied for the cleaning of historic silks.

Materials and Methods

Reagents and Materials

Fat-extracted oil-tea camellia seed cakes were provided from Xiangxi (Hunan, China) and ground into fine powder (<250 μm) in a mill before use. Ethanol (AR) was purchased from Hangzhou Gaojing Fine Chemical Industry Co., Ltd (Hangzhou, China). Vanillin (AR) and standard tea saponin (purity of 96 wt%) were purchased from Aladdin Chemistry Co., Ltd (Shanghai, China). 30 % (w/w) hydrogen peroxide and 95–98 % (w/w) sulfuric acid were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China).

Apparatus

Rotary evaporator (RE-52CS, Yarong Bio-instrument Co., Ltd. Shanghai, China); Vacuum drying oven (DZF-6020, Shanghai Jing Hong Laboratory Instrument Co., Ltd. Shanghai, China); High-speed centrifuge (TGL-16C, Shanghai Anting Scientific Instrument Factory, Shanghai, China); UV/Vis spectrophotometer (752S, Shanghai Lengguang Industrial Co., Ltd. Shanghai, China); Microwave extraction system (MAS-II, Shanghai Sineo Microwave Chemistry Technology Co., Ltd. Shanghai, China); Fourier transformed infrared spectroscopy (VECTOR222, BRUKER, Germany); High performance liquid chromatograph (1100series, Agilent, American).

Extraction Methods

Heat Reflux Extraction of Tea Saponin

Among the conventional extraction methods like maceration and stirring extraction, shaking extraction and ultrasonic extraction for the extraction of tea saponin, heat reflux extraction was adopted here to compare with MAE for its universality. In this experiment, heat reflux extraction was performed as Mandal [10] described, with some parameters changed. Oil-tea camellia seed cake powder (10 g) was extracted at 75 °C for 6 h under reflux with 200 ml of 85 % (v/v) ethanol aqueous solution in a 250-ml round-bottom flask heated in a water bath. After the extraction, the sample was centrifuged and the supernatant was evaporated for dryness under vacuum environment. Then the dried residue was decolorized by 30 % (w/w)

H₂O₂ aqueous solution for 1 h. Finally the dried and ground resultant powders (<250 μm) were ready for quantitative analysis.

Microwave-Assisted Extraction of Tea Saponin

Microwave-assisted extraction was performed in a closed microwave extraction system equipped with a magnetron of 2,450 MHz with a nominal maximum power of 1,000 W, a reflux unit, ten power levels, a time controller, a temperature sensor, an exhaust system, a liquid crystal display and a stirring device. Accurately weighed powder (10 g) of fat-extracted oil-tea camellia seed cake was mixed with ethanol aqueous solution in a 250-ml extraction vessel. Then the container was closed with a lid and a temperature sensor was inserted into the container to measure and control the internal temperature. The samples were irradiated with microwave energy under different experimental conditions, including changing microwave power, irradiation duration, temperature, ratio of solvent to material and aqueous ethanol concentration. After extraction, the samples were centrifuged, decolorized (30 % (w/w) H₂O₂ aqueous solution, 1 h), dried, ground (<250 μm) and submitted to quantitative analysis.

Vanillin Sulfuric Acid Colorimetry for Quantitative Analysis

Calibration Curve of Tea Saponin

The determination of the total content of tea saponin was performed as described by Xiang [21]. The standard curve which was used as the benchmark for the extraction yield determination was obtained as follows. Accurately weighed dried standard tea saponin (100.00 mg) was dissolved in 80 % (v/v) ethanol aqueous solution in a 100-ml volumetric flask. Standard tea saponin solutions with the concentration of 1 g/L and the amount of 0, 0.1, 0.2, 0.3, 0.4 and 0.5 ml were accurately transferred into a 5-ml test tube respectively. Then, 80 % (v/v) ethanol aqueous solutions were added to make the total volume being 0.5 ml. 0.5 ml vanillin anhydrous ethanol solution [8 % (w/v)] was added into these test tubes respectively. These test tubes were transferred into an ice water bath, and concentrated sulfuric acid solutions [4 ml, 77 % (w/w)] were added to the test tubes respectively, too. Then the tubes were transferred from the ice water bath into a water bath of 60 °C for 25 min. After being cooled to room temperature, with a blank solution as reference, the absorbency was scanned by using a UV/Vis spectrophotometer. Scanning results showed that the maximum adsorption was at 550 nm, so the absorbency at Vis 550 nm was determined with a quartz cuvette.

Regression gives the linear relationship:

$$C = 924.37A - 0.015(R^2 = 0.9973) \quad (1)$$

where C (mg/ml) is the concentration of tea saponin of solution for colorimetric analysis. A is the absorbance at Vis 550 nm. R is the correlation coefficient.

Determination of Total Extraction Yield

The accurately weighed tea saponin sample (100.00 mg) extracted above was dissolved in 80 % (v/v) ethanol aqueous solution in a 100-ml volumetric flask. The concentration of the sample was 1 g/L. 0.5 ml the solution was transferred into a 5-ml test tube. Then the absorbency of the sample was determined by the colorimetric method as described in 2.4.1 and the total extraction yield (Y) was calculated by Eq. (2) according to the standard curve.

$$Y = \frac{(A \times 924.37 - 0.015) \times m_1}{m_2 \times 1000} \quad (2)$$

m_1 (g) is the mass of the extracted tea saponin. m_2 (g) is the mass of oil-tea camellia seed cake powder. A is the same as in Eq. (1).

Results and Discussion

Analysis of Single Factor Experiments

The effects of five factors (microwave power, irradiation duration, temperature, ratio of solvent to material and aqueous ethanol concentration) were studied respectively to obtain the optimal parameters. In single factor experiments, only one parameter was changed, the other four parameters were constant. The constant parameters were set at a microwave power of 400 W, an irradiation duration of 4 min, a temperature of 60 °C, a ratio of solvent to material of 10 ml/g and an aqueous ethanol concentration of 70 % (v/v). The data around the parameter which corresponded to the highest yield were chosen as optimal for next orthogonal experiments. Each experiment was performed four times.

Effect of Microwave Power

Figure 1 shows the relationship between extraction yield and different microwave powers. In general, it shows that there is a sharp increase in extraction yield with the microwave power increasing from 100 to 400 W. However, no obvious variation in extraction yield is shown at power levels between 400 and 700 W. A quick decrease in extraction yield with increasing microwave power is observed for power levels higher than 700 W. A similar

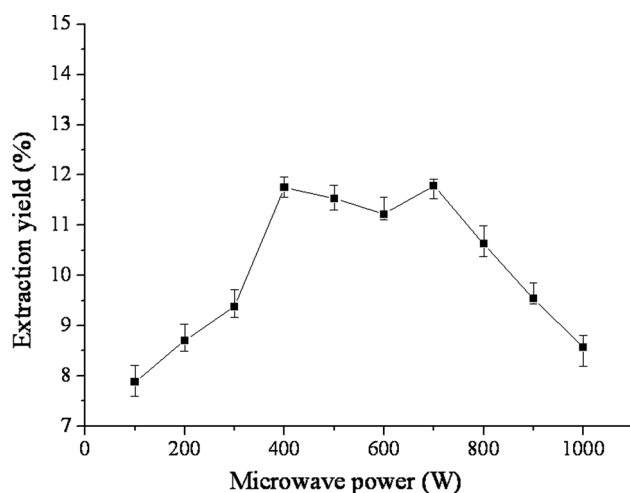


Fig. 1 Effect of microwave power on yield of tea saponin

observation was made in the MAE of flavonoids from *Saussurea medusa* maxim cultured cells [22].

During the process of MAE, microwave energy affects plant molecules directly by ionic conduction and dipole rotation which make the energy dissipate in a volumetric fashion inside the samples and solvents. Then molecular movement and heat are generated, and therefore the extractive is separated from the matrix [10, 23]. With the power increased from 100 to 400 W, more electromagnetic energy was transmitted to the solvents and samples, which resulted in more violent molecular movement and heating, and therefore more extractives were separated from the matrix. Thereby the extraction yield was gradually improved. At 400–700 W, evaporation of the solvent was speeded up by the rapid increase of electromagnetic energy and temperature resulting from the higher microwave power. Thus, although the microwave power was stronger, the solvent participating in the extraction system was limited, which consequently resulted in the extraction yield showing little change. At 700–1,000 W, the evaporation of the solvent was so violent under the influencing of high microwave energy that more and more solvent was released from the extraction system. As a result, the extraction yield showed a pronounced decrease. In conclusion, 400–700 W microwave power was considered as the optimum.

Effect of Irradiation Duration

The effects of irradiation duration on the yield of tea saponin are shown in Fig. 2. Three phases are observed in the extraction process. In the first phase (from 2 to 3 min), about 85 % of the total yield is obtained. In the second phase (from 3 to 5 min), the increase in the extraction yield tends to be slower. In the third phase (after 5 min), the

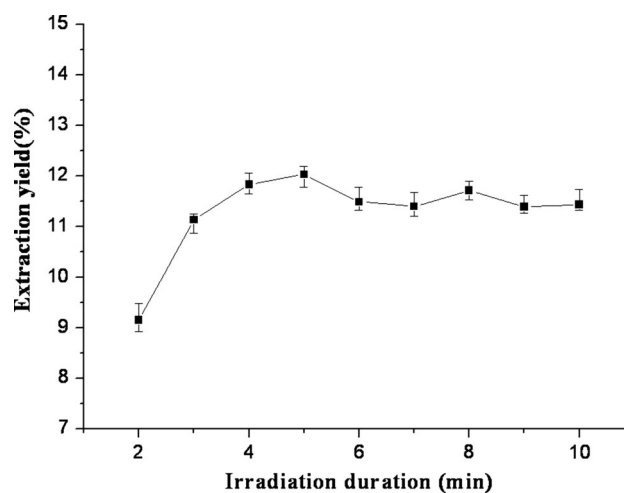


Fig. 2 Effect of irradiation duration on yield of tea saponin

yield of tea saponin decreases a little and there is no significant difference in yield with a longer duration. It seems that most tea saponins were extracted in the first 5 min. After 5 min, extraction almost ended and the extracted tea saponins were decomposed partially by being kept at high temperature for a long period of time. Similar tendencies were observed in the MAE of artemisinin [24] and of triterpenoid saponins from *Ganoderma atrum* [7]. Consequently, 4–7 min was chosen as the optimal irradiation duration.

Effect of Temperature

The effects of temperature on the yield of tea saponin were studied with eight levels at intervals of 5 °C from 40 to 75 °C. The results are shown in Fig. 3. In the range of 40–55 °C, the extraction yield improves with increasing temperature. On the one hand, molecular movement was enhanced with the increasing temperature. The inside of the plant matrix was easier immersed in solvent molecules. On the other hand, the dissolving capacity of the solvent was improved at higher temperatures [25]. Both of the factors favored the extraction of tea saponin. At 55–75 °C, the extraction yield decreases gradually with the increase in temperature. This is due to the rapid evaporation of solvent at higher temperatures and less solvent participated in the extraction system. From the above, 50–65 °C was considered optimal for the extraction of tea saponin.

Effect of Ratio of Solvent to Material

Figure 4 shows the relationship between extraction yield and the ratio of solvent (water–ethanol mixture) to the material. At 4–13 ml/g, the yield increases straightly with the increasing ratio of solvent to material. This is incurred

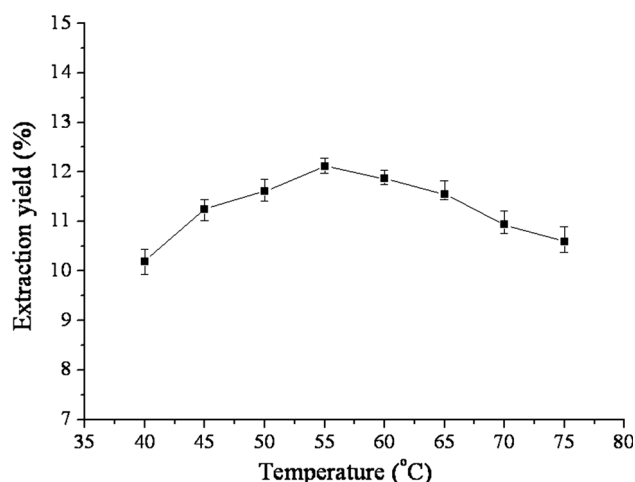


Fig. 3 Effect of temperature on yield of tea saponin

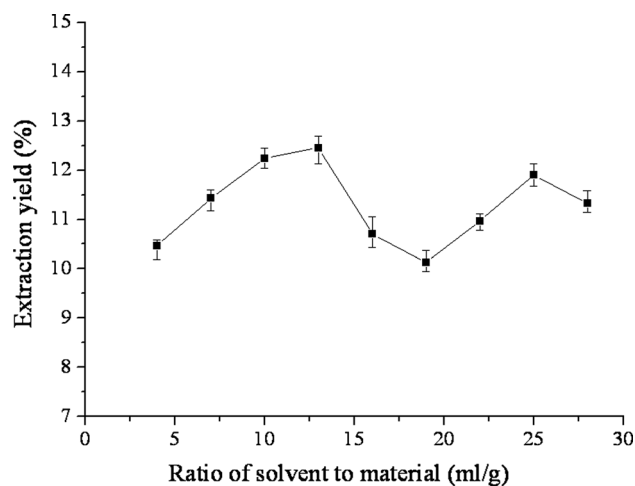


Fig. 4 Effect of ratio of solvent to material on yield of tea saponin

by the gradually increasing immersion of the sample in the solvent. At low ratios of solvent to material, the solvents were insufficient to immerse the samples. Therefore, the samples were not dissolved or heated enough to release tea saponin sufficiently. At higher ratios, the samples were fully dissolved by sufficient immersion. Consequently, microwave energy was well absorbed and functioned on the separation. At 13–19 ml/g, the yield drops sharply. This is probably due to insufficient stirring of the samples when solvent was in large volumes [25, 26]. As a consequence, some powders were still conglomerated with each other and could not be fully brought into contact with the solvent. Moreover, microwave energy was dispersed through larger volumes of solvent so that the samples could not absorb enough energy to break cells and release target substances. It is worth noting that at 19–25 ml/g the yield increases again. It is known that extraction is a process in which extractive molecules are separated with the matrix

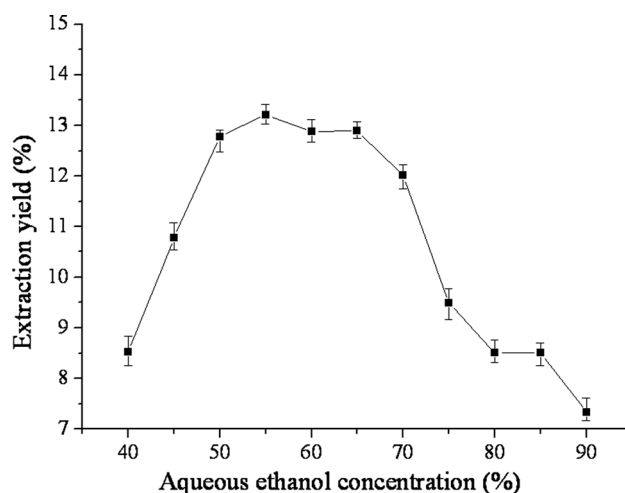


Fig. 5 Effect of aqueous ethanol concentration on yield of tea saponin

and diffused into the mixed solution. Large amounts of solvent might facilitate the diffusion of tea saponin molecules from the matrix to the solution, which resulted in more gathering of tea saponin and therefore a higher yield. However, when the amount of solvent was too large, the mixtures of sample and solvent were difficult to heat to the desired temperature in a short time. Consequently, the extraction yield decreased, as shown with more than 25 ml/g. In consideration of cost, within the range of 7–16 ml/g was considered to be the optimal ratio.

Effect of the Concentration of Aqueous Ethanol

The effects of different aqueous ethanol concentrations (11 levels at intervals of 5 % from 40 to 90 %) on yield are shown in Fig. 5. It shows that the yield of tea saponin is greatly influenced by the aqueous ethanol concentration. At 40–50 %, the increase in ethanol concentration resulted in a significant increase in extraction yield. This is probably due to the decrease in solvent polarity with the increasing ethanol concentration. It was found that high ethanol content will decrease the polarity of the mixture to a degree favorable for extraction [27]. At 50–65 %, the yield increases a little and starts to decrease. But there is no big change in the extraction yield. After 70 %, the extraction yield reduces rapidly. One possible reason is that a higher ethanol content would result in a lower solvent boiling point. As a consequence, ethanol evaporated extensively and the solubility in the solvent decreased. Another reason is that high ethanol content would facilitate the rapid solidification of impurities such as proteins, soluble polysaccharides, etc., which was not favorable for MAE. From the above results, 50–65 % was optimal aqueous ethanol concentration.

Analysis of Orthogonal Experiments

After the single factor experiments, the results of single effects of five factors on extraction yield were obtained. In order to determine the optimal parameters, a 4^5 orthogonal array was designed to investigate the synergistic effects of the five factors on extraction yield, as shown in Table 1. Each experiment was performed four times.

Table 2 gives the average extraction yields of sixteen groups, and the last five rows list the average extraction yields and the ranges of average extraction yields of each level for five parameters. The average extraction yield of Group 13 is 14.64 %, the highest of all. And its parameters are $A_4B_1C_4D_2E_3$ (aqueous ethanol concentration of 65 %, temperature of 50 °C, irradiation time of 7 min, microwave power of 500 W and ratio of solvent to material of 13 ml/g).

However, the results of orthogonal experiments should be analyzed to identify the optimal parameters.

In Table 2, the ranges of each level for five parameters are represented. The ranges of average extraction yields of each microwave power, irradiation time, temperature, solvent-to-material ratio, and aqueous ethanol concentration level are 0.36, 0.51, 0.39, 0.77 and 1.05 %, respectively. Therefore, the influence rank of each factor is as follows: aqueous ethanol concentration >ratio of solvent to material >irradiation time >temperature >microwave power. Aqueous ethanol concentration and ratio of solvent to material are the most two influential factors. Considering yield, efficiency and cost, $A_4B_1C_1D_1E_3$ (microwave power of 400 W, irradiation duration of 4 min, temperature of 50 °C, ratio of solvent to material of 13 ml/g, aqueous ethanol concentration of 65 %) was chosen as the final

Table 1 The factor levels in orthogonal experiment

Level	Factor A Aqueous ethanol concentration (%)	Factor B Temperature (°C)	Factor C Irradiation duration (min)	Factor D Microwave power (W)	Factor E Ratio of solvent to material (ml/g)
1	50	50	4	400	7
2	55	55	5	500	10
3	60	60	6	600	13
4	65	65	7	700	16

Table 2 The results of orthogonal experiment and range analysis

Group	A	B	C	D	E	Extraction yield (%)	Standard deviation ($n = 4$)
1	1	1	1	1	1	13.10	0.21
2	1	2	2	2	2	13.09	0.24
3	1	3	3	3	3	13.14	0.18
4	1	4	4	4	4	13.08	0.23
5	2	1	2	3	4	13.55	0.29
6	2	2	1	4	3	13.33	0.23
7	2	3	4	1	2	12.53	0.13
8	2	4	3	2	1	12.84	0.15
9	3	1	3	4	2	12.83	0.11
10	3	2	4	3	1	12.52	0.22
11	3	3	1	2	4	13.81	0.21
12	3	4	2	1	3	14.15	0.25
13	4	1	4	2	3	14.64	0.19
14	4	2	3	1	4	14.10	0.24
15	4	3	2	4	1	13.99	0.18
16	4	4	1	3	2	13.73	0.17
k1 (%)	13.24	13.66	13.63	13.61	13.25	/	/
k2 (%)	13.06	13.26	13.69	13.60	13.04	/	/
k3 (%)	13.33	13.37	13.23	13.23	13.82	/	/
k4 (%)	14.11	13.45	13.19	13.31	13.64	/	/
R (%)	1.05	0.39	0.51	0.36	0.77	/	/

Table 3 Comparison of MAE with heat reflux extraction method

Extraction method	Extraction time	Solvent consumption (ml)	Ethanol concentration (%)	Extraction yield (%)
MAE	4 min	130	65	14.73
Heat reflux extraction	6 h	200	85	12.88

optimal parameters. The average extraction yield under this condition is 14.73 %, which is slightly higher than the average extraction yield 14.64 % under $A_4B_1C_4D_2E_3$.

Comparison of MAE and Heat Reflux Extraction

As described above, a conventional extraction (heat reflux extraction) and MAE of tea saponin from oil-tea camellia seed cake (under the conditions of $A_4B_1C_1D_1E_3$) were conducted respectively and the relevant results are listed in Table 3. It can be seen that MAE has great advantages on extraction time, solvent consumption, ethanol concentration and extraction yield.

At present, conventional extractions like heat reflux extraction are widely used in many plants for active substance extraction. The two biggest disadvantages of conventional extractions are the high solvent and time consumption. Moreover, many conventional extractions use methanol and *n*-butanol as solvent. It is well known that methanol and *n*-butanol are much more toxic than ethanol to human being or the environment. Since extraction yield, efficiency, cost and most importantly, environmental friendliness and safety should be considered in the selection of extraction method, we believe that conventional extractions are not favorable in modern industry. MAE is a relatively new method, which is gaining popularity mainly by its efficiency. As shown in Table 3, the extraction time was reduced from 6 h (conventional

extraction) to 4 min (MAE). About 50 % ethanol was saved and the extraction yield was enhanced by about 14 % in MAE. These results prove that MAE is quite suitable for the extraction of tea saponin from oil-tea camellia seed cake. With the advancement in MAE technology, it may replace conventional extractions someday.

FTIR Comparison of the Extracted Tea Saponin and Standard Tea Saponin

FTIR spectra supplied information about molecular functional groups with which some chemical compounds can be identified. Figure 6 shows the FTIR comparison of the tea saponin extracted above and the standard tea saponin. Compared with the spectra of the standard tea saponin (Fig. 6a), characteristic peaks at $3,426\text{ cm}^{-1}$ attributed to the stretching vibration of O–H, $2,927\text{ cm}^{-1}$ presenting the antisymmetric stretching vibration of saturated $-\text{CH}_2-$, $1,717$ and $1,623\text{ cm}^{-1}$ indicating the existence of $-\text{CO}-$, $1,384\text{ cm}^{-1}$ contributed by the absorption of symmetrical formation vibration of $-\text{CH}_3$, and the band from 900 to $1,150\text{ cm}^{-1}$ presenting the absorption of C–O–C are all present in the spectra of the extracted tea saponin (Fig. 6b). Therefore, the resultant substance extracted above is tea saponin with compounds similar to the standard tea saponin.

HPLC Comparison of the Extracted Tea Saponin and Standard Tea Saponin

HPLC is a chromatographic technique used to separate the components in a mixture, to identify each component, as well as to quantify each component. Figure 7 shows the HPLC comparison of (a) standard tea saponin (b) extracted tea saponin and (c) non-discolored extracted tea saponin. There are two main peaks (Peak I, II) in the three curves (a, b, c). According to Liu's research [28], the HPLC curve of tea saponin contains two main peaks, similar to the Peak I and Peak II shown in Fig. 7, which present the characteristic peaks of theasaponin (effective component of tea saponin as a surfactant) and flavonoid (the coloring component for its yellow color), respectively. From the peak strength, which is the highest for Peak I, one finds that theasaponin is the main ingredient in the tea saponin extracted above and the standard tea saponin. The peak strength of Peak II in Fig. 7b is higher than that in Fig. 7a, which indicates that the amount of flavonoid is higher in

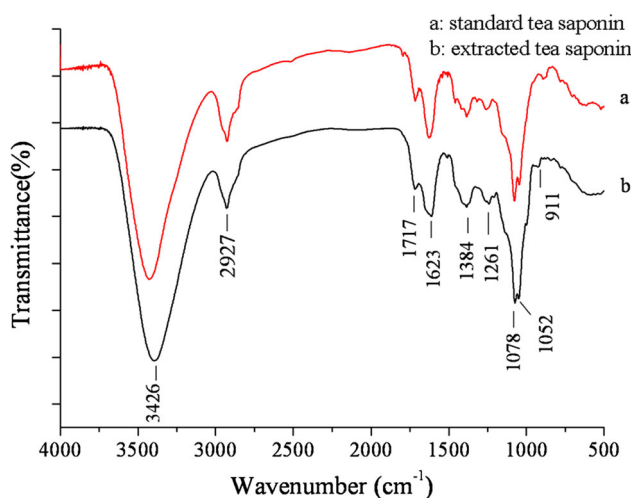


Fig. 6 FTIR comparison of (a) extracted tea saponin and (b) standard tea saponin

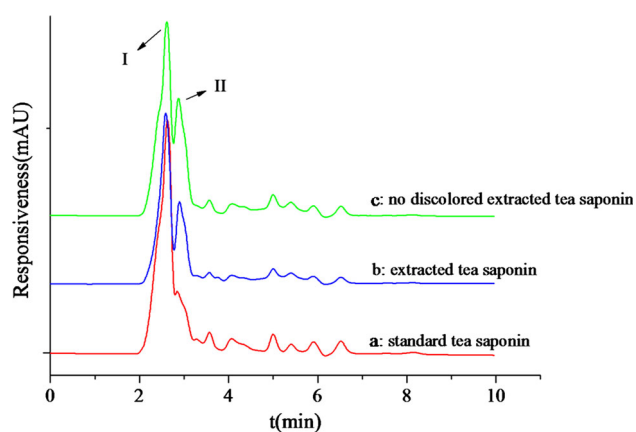


Fig. 7 HPLC comparison of (a) standard tea saponin (b) extracted tea saponin and (c) non-discolored extracted tea saponin

Table 4 Surface tension of different concentration tea saponin

Tea saponin concentration [% (w/w)]	Surface tension (Nm/m)
0.0	71.89
0.2	55.21
0.4	40.28
0.6	38.82
0.8	38.95
1.0	38.00
2.0	37.70
3.0	37.74

the extracted tea saponin than that of the standard tea saponin. But compared with the non-discolored extracted tea saponin, the amount of flavonoid is much lower, which is discernible from the much lower peak strength of Peak II in Fig. 7b compared with Fig. 7c. From these results, it can be said that the main ingredient of the extracted tea saponin is theasaponin, effective component of tea saponin as surfactant, with some loadings of flavonoid, the component giving it its yellow color. It needs to be further purified for application.

Application to the Cleaning of Historic Silks

Many cultural relics, especially those of contaminated historic textiles, need to be cleaned before further protection and conservation. But as we know, there is no specialized detergent that is designated for the cleaning of old textiles. In the cleaning of old textiles, it should be effective at stain removal and safe [29]. The safety includes no damage to the color, handle, strength etc. of the old textiles, and the friendliness to human beings and the environment, too. Therefore, it is important and urgent to develop effective and safe detergents for cleaning old textiles.

Therefore, the tea saponin extracted in this study was applied to the cleaning of old silks with further purification according to the description of Liu [30]. Table 4 shows the surface tension of purified tea saponin solutions at 20 °C. From this Table, one can see that the surface tension



Fig. 8 Photographs of a whole contaminated window shade silk and contaminated partial side, b before cleaning, and c after cleaning

changes little when the concentration of tea saponin is above 0.4 % (w/w). The critical micelle concentration (CMC) is considered to be an ideal concentration at which the surface tension of a surfactant solution is the lowest and the surfactant performs efficiently as detergent at this concentration [31]. Therefore 0.5 % (w/w) was chosen as the optimal concentration to prepare detergent here, considering the CMC of tea saponin is 0.5 % (w/w).

After evaluating the efficiency and safety of the extracted tea saponin on the cleaning of contaminated artificially aged silks and an old silk remnants, we applied it to the cleaning of historic silks. Figure 8 shows the cleaning effect of one piece of historic window shade silk (Qianlong Emperor Garden of Qing Dynasty, Palace Museum) contaminated by dust, paste, water stains, etc. It can be seen that most of the stains on the old silk were removed completely after cleaning; while the colors of the embroidery decorations and the ground cloth remain unchanged. What is worth mentioning is that the handle of the silk was improved after cleaning. It indicates that the tea saponin extracted in this study is effective and safe for cleaning old silk. Tea saponin could be a candidate for a detergent for the cleaning of contaminated historic textiles.

Conclusions

Tea saponin containing compounds similar to a standard tea saponin was extracted by MAE from oil-tea camellia seed cake. Compared with conventional extraction, the MAE method saves much time and solvent and is more environmentally friendly with a higher efficiency. The extracted tea saponin has been effectively applied for the cleaning of contaminated old silks.

Acknowledgments This work was financially supported by Zhejiang Culture Relics Protection Fund (No. 2011202 and 2010264).

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