

Enhancement of Oil Extraction from Sumac Fruit using Steam-Explosion Pretreatment

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Abstract Steam explosion, in which materials are crushed through rapid decompression after high-pressure steaming, has been widely used for biomass pretreatment. In this study, we used steam-explosion pretreatment (SEP) for oil extraction from sumac fruit. SEP reduced particle size and formed fissures and micropores on the fruit. The kinetics of the extraction process indicates that under optimal conditions (steam pressure 1.5 MPa, residence time 3 min), the oil yield at equilibrium increased to 16.04%, approximately fourfold higher than that of the raw sample. The mass transfer coefficient of the oil extraction decreased as a result of SEP. In addition, the acidity of the extracted oil decreased and peroxide value increased in the steam-exploded sample compared with that of the raw material. Few changes in fatty acid composition were observed, with low palmitic acid content and high linoleic acid composition. These results suggest that SEP improved the yield of oil extraction from sumac fruit and affected oil quality.

Keywords Steam explosion · Pretreatment · Sumac fruit · Oil extraction · Kinetics

Introduction

Sumac is the common name for the genus *Rhus* comprised of over 250 species in the family *Anacardiaceae*. These plants are wild arbors that are prevalent in temperate and

tropical regions worldwide. Millions of tons of sumac fruit are produced annually in China, but this yield has yet to be exploited commercially. The oil of sumac fruit is used as an edible oil in some areas, although there has been no report proving its edibility. Sumac fruit contains 12.0–20.8% oil, with linoleic acid ($C_{18:2}$) being the predominant fatty acid (47.4%) [1]. As a valuable unsaturated fatty acid, linoleic acid is thought to be capable of lowering LDL cholesterol and reducing the risks associated with heart diseases.

Various new technologies, including ultrasonic [2] and supercritical fluid extraction [3], have been used for extracting oil from plants to increase extraction yield and enhance extract quality. However, many operations prior to extraction, such as grinding [4], cooking [5], and drying [6], also affect oil yield and quality. Therefore, the pretreatment of oilseeds is an essential process.

Steam explosion is the most commonly used method for the pretreatment of lignocellulosic materials [7–9]. The physicochemical treatment steams materials with high-pressure saturated steam for several seconds to a few minutes, and then exposes these materials to atmospheric pressure. Steam pressure and residence time are the two main parameters of steam explosion. Higher steam pressure contributes to obtaining a smaller particle size. Residence time affects the softening degree of materials and permeation of steam. This technology has been used for the extraction process in studies such as that of Kurosumi et al. [10], who reported that steam explosion followed by hot water and methanol extractions is a highly effective method for extracting antioxidants from tissues of *Sasa palmata*. The application of steam-explosion pretreatment (SEP) in oil extraction, however, has not yet been reported.

Steam explosion, coupling cooking, and breaking may be an effective pretreatment method for oil extraction from

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oilseeds. This study aims to investigate the feasibility of using SEP to enhance oil extraction from sumac fruit. The effects of SEP on the particle size and microstructure of the fruit were investigated. The kinetics of the oil extraction process from steam-exploded sumac fruit were studied, and the quality of extracted oil was analyzed.

Materials and Methods

Materials

Sumac (*Rhus chinensis* Mill.) fruit from Anhui Province was hand cleaned and dried in a shady, well-aired environment for 15 days, and then packed in paper bags and stored in a dark and dry place at room temperature until use. The prepared fruit contained 5% moisture content. Different parts (pulp, endocarp, and seed) were manually separated and weighed. The oil content of the entire fruit and its isolated parts was determined by Soxhlet extraction.

Steam Explosion

The steam-explosion vessel (4.5 l, Weihai Automatic Control Reactor Ltd., Weihai, China) consisted of a steam generator, high-pressure reactor, receiver, and condenser [11]. Sumac fruit (about 500 g) was pretreated at different pressure levels (1.3 and 1.5 MPa) for 3 or 5 min by using saturated steam; the process was then terminated by explosive decompression [8, 12]. The steam-exploded fruit was dried at 60 °C for 12 h to about 3% moisture, ground with a mortar and pestle, sieved into four different particle size ranges (2–3, 0.85–2, 0.25–0.85, and <0.25 mm), and weighed. The particles were considered to be spherical, and the average particle size was calculated by randomly selecting 100 particles and using a microscope (Olympus BX41, Olympus Corp., Tokyo, Japan).

Scanning Electron Microscopy

Microstructure observations of raw and steam-exploded sumac fruit were carried out via scanning electron microscopy (SEM, JSM-6700F, JEOL, Tokyo, Japan), conducted under vacuum at 5.0 kV accelerating voltage.

Oil Extraction Using Solvent

Batch extraction was conducted in a capped flask at 25 °C using petroleum ether (boiling point range, 30–60 °C) as the solvent with a solids-to-solvent ratio of 1:10 (m/v). Samples were extracted every 20 min for up to 120 min. The miscella, containing oil and solvent, was poured into a 500-ml flask, and a rotary evaporator was used to remove

the solvent. The residual crude oil extracts were collected, weighed, and stored in sealed amber glass vials at –20 °C for analysis. Oil yields (wt%) at different times were calculated on a dry-weight basis according to Eq. 1.

$$\text{Oil yield (\%)} = \frac{m}{M} \times 100 \quad (1)$$

where m is the extracted oil mass (g), and M is the dry mass of the sample (g).

Total oil contents were determined by Soxhlet extraction after the samples were ground by using an electrical mill (XJZF-1A-type plant grinder, Yinhe Instrument Factory, JiangYan, China) through a 1-mm screen. The weight of the fruit used for the extraction was 10 g.

Kinetics Model of Oil Extraction

Oil extraction using a solvent involves two simultaneous physical processes [13–15]: *solution extraction* for oil extraction from ruptured cells and *diffusion extraction* from intact cells. A portion of the oil is quickly derived from broken oil-bearing cells, whereas oil removal from the intact cells, which requires osmosis, takes a much longer time. Thus, oil extraction is controlled mainly by diffusion in most cases. In this research, the kinetics of the oil extraction was fitted to the modified equation of So and Macdonald [14].

$$C_t = C_e(1 - \exp(-kt)) \quad (2)$$

where C_t was the oil yield (wt%) at time t (min), C_e was the oil yield at equilibrium (wt%), and k is the mass transfer coefficient for the extraction process (min^{-1}). The values of the oil yield at equilibrium C_e and mass transfer coefficient k were calculated numerically with a non-linear least squares fitting method using Origin 8.0 (OriginLab Corporation, Northampton, MA).

Analysis of Extracted Oil

Acid and Peroxide Values

The acidity and peroxide value of the extracted oil were determined according to the standard AOCS methods F 9a-44 and Cd 3-25, respectively [16].

Fatty Acid Composition

The methyl esters of crude oil were prepared according to the method of Peng and Chen [17]. Fatty acid methyl esters (FAMES) were determined by using a gas chromatograph (Agilent 4890D) equipped with an HP-INNO Wax capillary column (30 m × 0.25 mm id, $df = 0.25 \mu\text{m}$) and a flame ionization detector. Oxygen-free nitrogen was used

as the carrier gas. Other conditions were as follows: initial oven temperature, 150 °C; ramp rate, 20 °C/min; final temperature, 250 °C; injector temperature, 260 °C; detector temperature, 260 °C. FAMES were identified by comparing their relative and absolute retention times with those of Sigma standards, and the fatty acid composition was reported as a relative percentage of the total peak area. All experiments were carried out in triplicate sets. The results reported are the averages of the three sets of experiments with a standard deviation.

Results and Discussion

Oil Content of Sumac Fruit

Sumac fruit is a drupe composed of a pulp (exocarp and mesocarp), endocarp, and seed. Despite the papery pulp that can easily be detached from the endocarp, the seed is provided sufficient protection by the lignified endocarp. Figure 1 shows the oil content and weight percentage of the parts of sumac fruit. The seed has the highest oil content (25.14%), followed by the pulp with 19.70%. The lowest content (10.74%) was observed in the endocarp; however, the weight percentage of the endocarp was larger than those of the pulp and the seed.

Effect of SEP on the Structure of Sumac Fruit

Reduction in particle size is one of the primary modifications that occur during steam explosion. Figure 2 shows the effects of SEP conditions on the particle size of sumac fruit. The diameter of a raw sumac fruit is 2–3 mm. When exploded under low-steam pressure for a short residence time (1.3 MPa, 3 min), the sumac fruit was still larger than 2 mm because the endocarp remained intact. Particle size decreased with the increasing intensity of SEP. For the sample pretreated at 1.5 MPa for 5 min, almost no intact

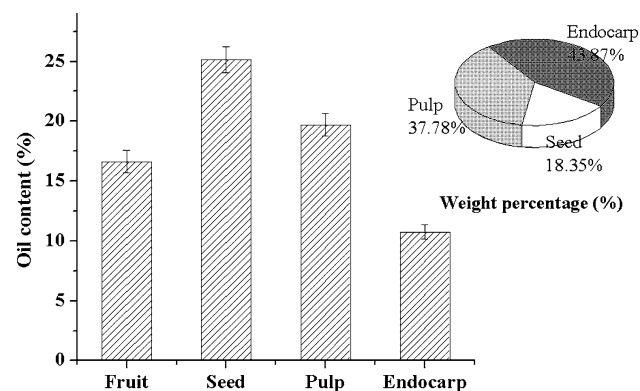


Fig. 1 Oil contents and weight percentages of the parts of sumac fruit

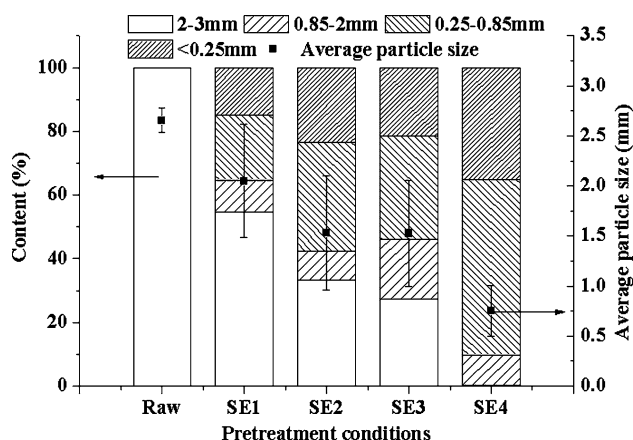


Fig. 2 Particle size of sumac fruit steam-exploded under different conditions. The column is the content percentage for different particle sizes. Scatter is the average particle size in different pretreatment conditions. Raw untreated sumac fruit; SE1 sumac fruit steam-exploded at 1.3 MPa for 3 min; SE2 1.3 MPa 5 min; SE3 1.5 MPa 3 min; SE4 1.5 MPa 5 min

endocarp was found, and the particle size larger than 2 mm accounted for only 0.21% of all particle sizes observed.

Another modification during steam explosion is the expansion of micropores [18]. Figure 3 shows the scanning electron micrographs (SEMs) of the endocarp of steam-exploded and raw sumac fruit. SEP caused marked structural changes, fissures, and cavities (Fig. 3a) on the endocarp. By contrast, the endocarp of the raw sumac fruit exhibited a regular and compact shape, and no cavities were observed (Fig. 3b).

Kinetics of Oil Extraction from Steam-exploded Sumac Fruit

Both reduction in particle size and expansion of micropores can increase the surface area of materials to promote contact with solvents and materials. Thus, SEP was expected to enhance oil extraction from sumac fruit. Figure 4 shows the effect of steam-explosion conditions on oil extraction yield, and the experimental data were fitted to the modified mathematical model of So and Macdonald [14]. Good fits were obtained as shown by the high values of the coefficients of determination ($0.99 < R^2 < 1.00$) given in Table 1. Steam-exploded samples exhibited higher oil yields than did the raw sample, and oil yield was positively correlated with SEP intensity when all other factors remained constant. Under 1.5 MPa and 3 min, the oil yield reached 16.04% at equilibrium, about fourfold higher than that of the raw sample. At this point, raising steam pressure or prolonging retention time would not considerably improve oil yield, and additional energy would be wasted. Therefore, 1.5 MPa and 3 min were chosen as the optimal conditions of SEP for oil extraction from sumac fruit.

Fig. 3 Scanning electron micrographs of **a** steam-exploded and **b** raw sumac fruit endocarps

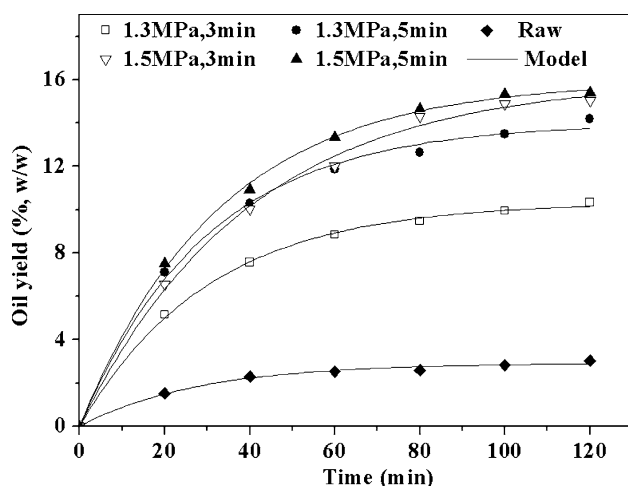
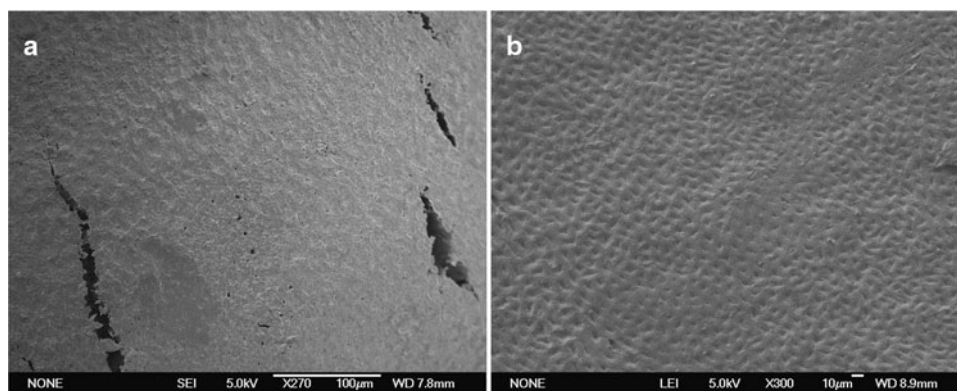


Fig. 4 Effect of the steam-explosion conditions on oil extraction yield of sumac fruit. Extraction solvent, petroleum ether; fruit-to-solvent ratio (g/ml), 1:10; temperature, 25 °C

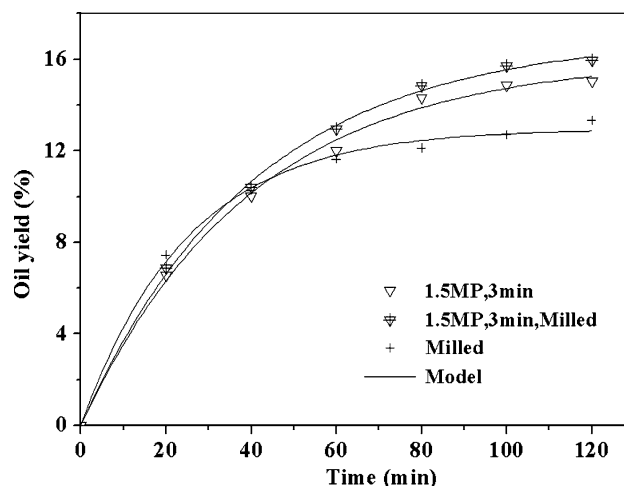


Fig. 5 Comparison of kinetics of the oil extraction from steam-exploded and milled sumac fruit. Extraction solvent, petroleum ether; fruit-to-solvent ratio (g/ml), 1:10; temperature, 25 °C

Table 1 Values resulting from fitting kinetic data of oil extraction to modified model of So and Macdonald: $C_t = C_e(1 - \exp(-kt))$

Pretreatment condition	C_e	k	R^2
Raw	2.92 ± 0.08	0.0358 ± 0.0035	0.9902
1.3 MPa, 3 min	10.34 ± 0.11	0.0331 ± 0.0012	0.9987
1.3 MPa, 5 min	14.00 ± 0.26	0.0333 ± 0.0021	0.9961
1.5 MPa, 3 min	16.04 ± 0.38	0.0251 ± 0.0017	0.9964
1.5 MPa, 5 min	15.95 ± 0.18	0.0304 ± 0.0011	0.9987
1.5 MPa, 3 min, milled	16.99 ± 0.27	0.0247 ± 0.0011	0.9984
Milled	12.97 ± 0.21	0.0403 ± 0.0025	0.9959

Moreover, a reduction in the mass transfer coefficient (6.98–29.89%) is observed in SEP.

Sumac fruit steam-exploded under the optimal conditions and the raw sample were separately milled through a 1-mm screen to compare steam-explosion and milling. The kinetics of the oil extraction process was then analyzed (Fig. 5). The values are shown in Table 1. The oil yield at equilibrium of the steam-exploded-milled sample was

Table 2 Acidity and peroxide value of oils extracted from steam-exploded sumac fruit

Pretreatment condition	Acidity (mg KOH/g oil)	Peroxide value (mmol/kg oil)
1.3 MPa, 3 min	5.8 ± 0.2	5.6 ± 0.4
1.3 MPa, 5 min	5.3 ± 0.6	7.7 ± 0.3
1.5 MPa, 3 min	5.3 ± 0.7	6.8 ± 0.5
1.5 MPa, 5 min	5.1 ± 0.2	12.9 ± 0.2
Raw	6.7 ± 0.5	5.6 ± 0.1
Crude soya bean oil	≤4.0 ^a	≤7.5 ^a
Finished product of soya bean oil	≤3.0 ^a	≤6.0 ^a

^a Standards in China

slightly higher than that of the steam-exploded sample (5.92%), but was much higher than that of the milled sumac fruit (30.99%). Little difference in the mass transfer coefficient between steam-exploded and steam-exploded-milled samples was observed, but their mass transfer

Table 3 Fatty acid composition (%) of oils extracted from steam-exploded sumac fruit

Pretreatment condition	C16:0 (Palmitic)	C18:0 (Stearic)	C18:1 (Oleic)	C18:2 (Linoleic)	C18:3 (α -Linolenic)	Others
1.3 MPa, 3 min	30.90 \pm 1.15	2.73 \pm 0.07	11.54 \pm 0.59	47.75 \pm 1.85	2.47 \pm 0.16	4.61 \pm 0.32
1.3 MPa, 5 min	29.70 \pm 1.62	2.81 \pm 0.08	12.27 \pm 0.76	50.83 \pm 2.51	2.71 \pm 0.13	1.68 \pm 0.08
1.5 MPa, 3 min	29.60 \pm 0.72	2.87 \pm 0.01	12.50 \pm 0.09	50.44 \pm 1.65	2.83 \pm 0.05	1.76 \pm 0.02
1.5 MPa, 5 min	28.61 \pm 0.92	2.84 \pm 0.10	12.61 \pm 0.68	51.30 \pm 1.92	2.83 \pm 0.25	1.81 \pm 0.20
Raw Fruit	32.76 \pm 1.25	2.86 \pm 0.05	12.25 \pm 0.34	47.78 \pm 1.12	2.73 \pm 0.22	1.62 \pm 0.09
Pulp	39.98 \pm 1.08	3.90 \pm 0.20	14.36 \pm 0.45	34.48 \pm 0.03	3.78 \pm 0.02	3.50 \pm 0.01
Endocarp	12.73 \pm 0.27	2.83 \pm 0.06	12.31 \pm 0.04	67.88 \pm 1.83	1.36 \pm 0.03	2.89 \pm 0.02
Seed	9.73 \pm 0.21	2.44 \pm 0.14	11.13 \pm 0.38	74.14 \pm 1.20	1.42 \pm 0.09	1.14 \pm 0.06

coefficients were both lower than that of the milled sample (by 37.72 and 38.71%, respectively).

SEP (coupling, cooking, and breaking) increased the oil yield at equilibrium, but reduced the mass transfer coefficient. A similar result was reported by So and Macdonald [14], who found that a smaller sample size results in higher oil yield and cooking results in increased oil yield at equilibrium and decreased kinetic coefficient.

Oil extraction using a solvent involves two simultaneous physical processes: solution and diffusion. SEP disrupted some of the cells in the fruit and released more oil to the surface of the fruit. This phenomenon increased the solution stage equilibrium level, but decreased the diffusion stage equilibrium level. Given that the extraction process is generally controlled by the diffusion stage, the mass transfer coefficient was decreased with SEP.

Effects of SEP on Oil Quality

The high temperature of 190–200 °C, corresponding to the steam pressure of 1.3–1.5 MPa employed in SEP, might affect the physicochemical characteristics of the extracted oil. As shown in Table 2, acidity decreased, but the peroxide value increased with SEP. Theoretically, oil extracted from oilseeds pretreated under high temperatures is prone to hydrolysis [19], leading to a higher acid value than that of the untreated sample. The lower acid values of the oil from steam-exploded samples probably stemmed from the short retention time and evaporation of free fatty acid in the oil at the moment of explosion. While oxidation of certain materials in sumac fruit oil led to high peroxide values, SEP might have accelerated the reaction. Although the acid and peroxide values obtained with SEP were relatively slight compared with the values of the raw sample, these two values did not satisfy the standards for oil products in China (Table 2). Accordingly, alkali refining and clay absorption will be required to lower the acid and peroxide values of the oil.

Table 3 shows the effect of SEP on the fatty acid composition of oil from sumac fruit. The oil composition

of the steam-exploded sumac fruit was similar to that of the raw sample, except that the former had lower palmitic acid content (5.68–12.67%) and higher linoleic acid content (–0.06 to 7.37%). These results may have been caused by the SEP's enhancement of the oil extraction from the endocarp, which had low palmitic acid composition and high linoleic acid content compared with the entire sumac fruit.

Conclusion

SEP increased oil yield at equilibrium, but decreased the mass transfer coefficient according to the kinetics of the oil extraction of sumac fruit. Moreover, the quality of the extracted oil did not change dramatically. This work suggests that SEP effectively extracts oil from sumac fruit.

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