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Extraction of sesamin from sesame oil using macroporous resin

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ABSTRACT

Sesamin is drawing research attention, due to its effects on the various body regulators. Currently there are two methods employed to separate sesamin: solvent extraction and steam stripping, but both methods have disadvantages in large-scale manufacturing systems. An innovative method for sesamin extraction from sesame oil has been unveiled in this paper, which employs macroporous resin as an adsorbing surface. The final product, sesamin crystal, has been obtained by crystallization of the desorption product. The concentration of sesamin in the desorption product was 9.7%, nearly 20-fold greater than in the starting sesame oil. After further refining, the concentration of sesamin in the final crystalline product reaches 76%. The procedure described in this paper demonstrates that a high concentration of sesamin can be obtained by employing resin adsorption.

1. Introduction

Lignans, a class of secondary plant metabolites produced by oxidative dimerization of two phenyl propanoid units are major components of unsaponifiable matter in sesame (Sukumar et al., 2008). Sesamin is one of lignans in sesame, which accounts for over half of the amount of lignans corresponding to 0.07–1.13 weight percent in sesame (Fukuda et al., 1988; Tashiro et al., 1990). A series of biological activities of sesamin have been reported including regulating lipid and alcohol metabolism (Yoshinobu, 2004; Yoshinobu et al., 2005), antihypertensive effect (Nakano et al., 2004), cholesterol-lowering activity (Peñalvo et al., 2006), anti-inflammatory effect (Utsunomiya et al., 2003) and inhibition of breast cancer (Liu et al., 2006). These studies on sesamin are drawing research attention.

However, ways to separate sesamin from sesame oil are still not good enough. Currently there are methods employed: one uses organic solvents to extract sesamin from sesame oil (Omohundro and Fanto, 1949), while the other employs a steam stripping process (Ozaki and Hoshii, 1993). The former method has limitations: (1) low extraction yield of sesamin (2) the necessary inclusion of an extra saponification step and (3) results in a low value-added by-product (recovered sesame oil) containing a residual solvent that is difficult to remove completely. In the latter method, intense energy consumption is serious drawback and additional deodorization device is required to eliminate off-flavors, free aliphatic acids, etc. for edible oil.

A new method is proposed here. Macroporous resin can be used to adsorb sesamin from sesame oil. This adsorption material is commonly used in extraction from aqueous phase (Kim et al., 2007; Ou et al., 2007; Kan et al., 2009), but in this study, we used it to extract sesamin from oil phase by selecting the right type of resin. This technology had advantages of high extraction efficiency and low cost. In addition, the quality of sesame oil after sesamin extraction was assured and could meet Grade 2 criteria (GB8233-87, Chinese Standards, Table 1) because of pharmaceutical-grade macroporous resin employed, which also rendered the recovered sesame oil fit for human consumption. The resin could be regenerated after washed with acetone, distilled water and ethanol, and the solvents also could be reused after distillation.

2. Materials and methods

2.1. Materials

Sesame oil (hot moulding) was obtained from Shanghai Pansun Sesame Research Institute (Shanghai, China). Sesamin standard was purchased from Sigma Chemical Company (St. Louis, USA). Macroporous resin 1 (MR1) and Macroporous resin 2 (MR2) were obtained from Rohm and Haas Company (USA). Macroporous resin 3 (MR3) was obtained from Mitsubishi Chemical Corporation (Japan). Macroporous resin HZ-801, HZ-816 and HZ-818 were obtained from Shanghai Huazhen Sci. and Tech. Co., Ltd. (Shanghai, China). Manufacturers' information regarding the characteristics of the resins are reported in Table 2.

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Table 1
Specifications of Grade 2 criteria for sesame oil.

Characteristic of smell	Clarity	Color (Lovibond Tintometer with 1 inch cell)	Moisture and volatile matter (%)	Impurity (%)	Acid value (mg NaOH/g)	Saponification value (%)
No off-flavor	Slight visible turbidness is allowed	Yellow 70, red $\leqslant$ 12.0	$\leqslant$0.20	$\leqslant$0.20	$\leqslant$4.0	$\leqslant$0.03

Table 2
Chemical and physical properties of selected resins.

Resin	Matrix	Surface area (m^2/g)	Average pore diameter (Å)	Polarity
MR1	Phenol–formaldehyde	200	600	Moderately polar
MR2	Aliphatic ester	500	450	Moderately polar
MR3	Styrene–divinylbenzene	590	520	Non-polar
HZ-801	Styrene–divinylbenzene	–[a]	–	Slightly polar
HZ-816	Styrene–divinylbenzene	–	–	Slightly polar
HZ-818	Styrene–divinylbenzene	–	–	Non-polar

[a] Not given.

2.2. Analysis optimization of sesamin in sesame oil

Kamal-Eldin et al. (1994) reported a HPLC method to determine concentrations of lignans and lignan glycosides in sesame. But the accuracy was affected by the baseline shift and these methods were not satisfactory. Therefore the optimal condition to determine concentration of sesamin in sesame oil by HPLC was first studied.

SHIMADZU HPLC (Kyoto, Japan) was equipped with SCL-10Avp system controller, SPD-10Avp UV–VIS detector, LC-10Advp pump, CTO-10Asvp Column oven and Class-VP software. The detailed parameters to measure concentration of sesamin in sesame oil by HPLC were optimized as follows: VP-ODS column (150 mm × 4.6 mm) was used at 30 °C; methanol–water (74:26, v/v, volume ratio) was used as mobile phase at the flow rate of 1.0 ml/min; the detecting wavelength for sesamin was 286 nm.

2.2.1. Establishing calibration curve

Sesamin standard samples (6.25 mg, 12.5 mg, 25.0 mg, 37.5 mg, 50.0 mg and 62.5 mg) were weighed into six 50 ml volumetric flasks and dissolved in methanol to scale, respectively. All solutions were blended by ultrasonic cleaning machine (KS-150D, Ningbo Haishu Kesheng Ultrasonic Equipments Co., Ltd., Ningbo, China) for 30 min and filtered through 0.45 μm nylon filter. A volume of 10 μl of the above-stated sesamin solution was injected into the HPLC column. Then, the calibration curve was established.

2.2.2. Evaluating solvents which dissolve sesame oil

Another improvement in this study was the solvent selection for sample injection. 0.1 g sesamin standard was dissolved in 25 g sesame oil (the sesamin concentration in original sesame oil was known). Then eight samples containing 1.00 g of the aforementioned mixed sesamin oil were dissolved in 10 ml *n*-hexane, cyclohexane, benzene, mixture of *n*-hexane/chloroform (2:1, v/v), chloroform, acetic ether, butanol and acetone, respectively. A volume of 10 μl of the aforementioned solution was injected into the HPLC column and the sesamin concentration in sesame oil was determined. Each experiment was replicated thrice. The recovery and relative standard deviation (RSD) were calculated for each solvent to evaluate their dissolvability. All the sesamin concentrations reported below were determined by HPLC.

2.3. Extraction technique of sesamin

The schematic diagram for the extraction of sesamin is illustrated in Fig. 1. Sesame oil was mixed with macroporous resin for adsorption. The mixture was loaded into a glass chromatographic column after adsorption. Sesame oil was driven out by a

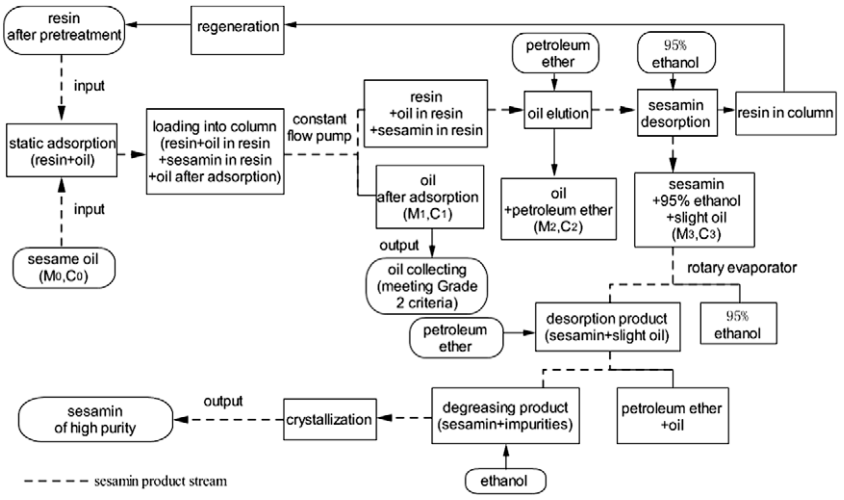

Fig. 1. Flow chart of the sesamin extraction technique (The M's and C's included in the figure were symbols in Eq. (2).).