

Study on supercritical fluid extraction of solanesol from industrial tobacco waste

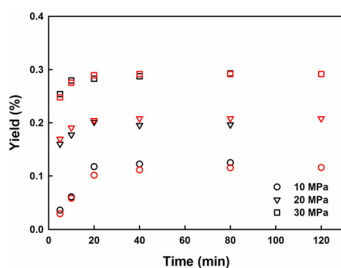


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GRAPHICAL ABSTRACT

Extraction yield of solanesol at pressures of 10 MPa, 20 MPa and 30 MPa, temperatures of 80 °C, 1.0 LPM CO₂ flow rate and 175 μm mean particle size; black: experimental data, red: model correlation.



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ABSTRACT

Extraction of solanesol from industrial tobacco waste using supercritical CO₂ was studied. The extraction yield was obtained at various pressures, temperatures, CO₂ flow rates and mean particle diameters. The content of solanesol in extract was determined by HPLC analysis. The broken and intact cell model was successfully applied to both the tobacco extraction kinetics and solanesol extraction kinetics. The internal mass transfer coefficients were computed for both extracts and solanesol at various experimental conditions. The extraction rate of solanesol was found higher than that of tobacco extract, indicating that shorter extraction time resulted in higher selectivity of solanesol. Furthermore, a pretreatment with a solvent of n-hexane:95% ethanol at 4:6 volumetric ratio was tried at a selected experimental condition to improve the selectivity of solanesol. A final product with solanesol content of 0.44 was obtained. Consequently, this study provides a new route for generating solanesol from industrial tobacco waste.

1. Introduction

Tobacco (genus: *Nicotiana*, family: *Solanaceae*) is one of the widely planted crops in the world. About 40% of the world's total tobacco production are produced and consumed in China, where tobacco plantation and manufacturing generate more than 200 million tons of tobacco waste annually [1]. Tobacco waste such as defective tobacco leaves and discarded picadura from plantation and production have

resulted in a serious environmental contamination [2]. The utilization of tobacco waste can help promote economic effectiveness of tobacco manufacturing and lighten the burden on environment, thus is an important task for researchers and tobacco industry.

Solanesol, a polyisoprenoid alcohol with several non-conjugated double bonds, is mainly accumulated in solanaceous crops (tomatoes, potatoes, eggplant, bell/chili peppers and tobacco), among which tobacco leaves are confirmed to hold the highest solanesol content

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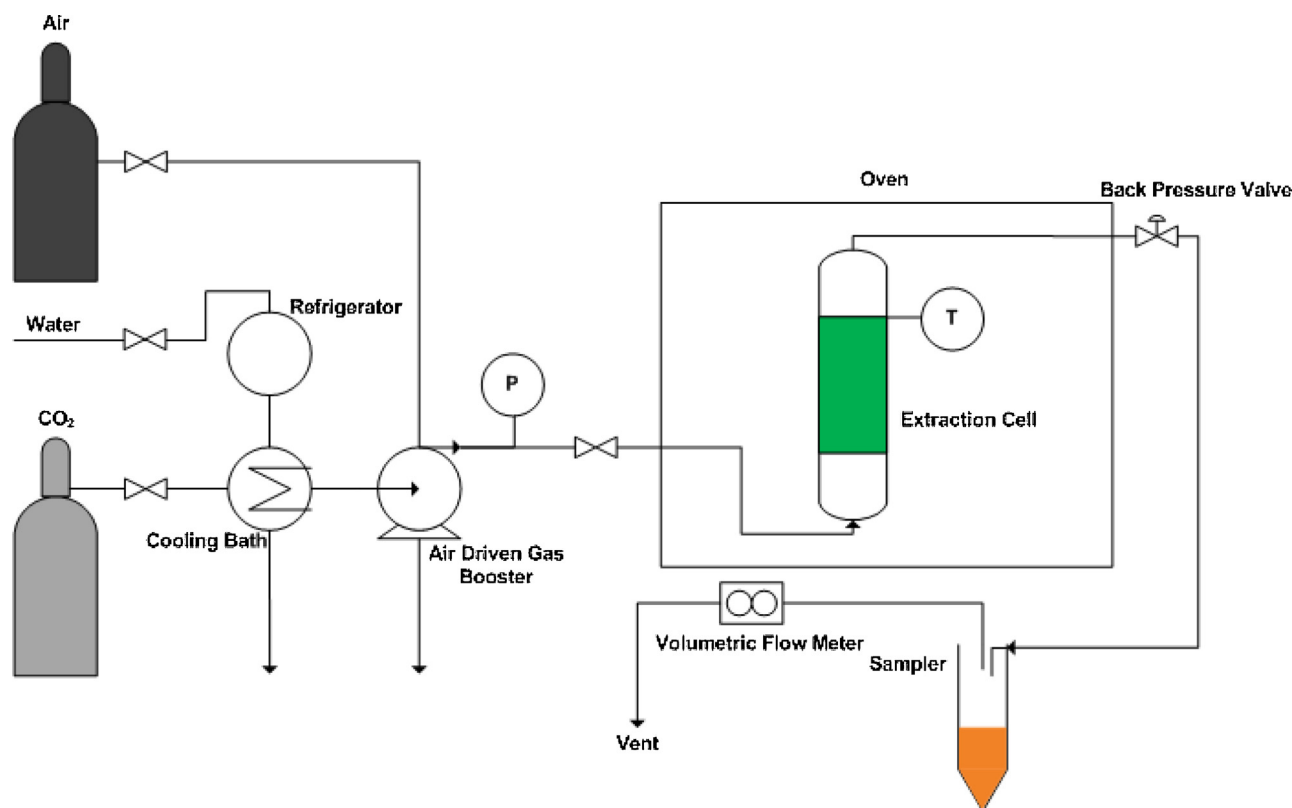


Fig. 1. Schematic diagram of supercritical CO₂ extraction system.

(0.45%) [3]. It is difficult to synthesize solanesol chemically due to its complex structure. Therefore, tobacco leaves are the main source of solanesol. Since the compound was first extracted from tobacco leaves by Rowland et al. in 1956 [4], it has been used in pharmaceutical industry as a pharmaceutical intermediate to synthesize coenzyme Q10 [5]. The compound was discovered to be effective in several bioactivities and medicinal applications such as anti-oxidant [6], anti-bacterial [7], anti-inflammatory [8], anti-fungal [9], anti-ulcer [10], anti-tumor [11] and wound healing [12].

Various extraction paths have been established to isolate solanesol from tobacco leaves. The most commonly employed extraction method is heat-reflux extraction [13]. Several new extraction methods have been developed in recent decades, including microwave assisted extraction [14], ultrasound-assisted extraction [15], solid phase extraction [16], column-chromatographic extraction [17] as well as supercritical fluid extraction (SFE) [9,18–21]. To improve the separation performance, organic solvents such as alcohols and alkanes are often used to enhance solanesol extraction selectivity considering the solvent interaction with solanesol's long chain alkyl groups [14,22].

SFE with CO₂ is broadly applied in food, pharmaceutical and cosmetic industry [19,23–25]. In tobacco industry, this technology is used to generate tobacco oils and to eliminate nicotine from tobacco [21,26]. Supercritical CO₂ has advantages over conventional method because of its low operating temperature and low cost [27]. However, pure CO₂ sometimes is unable to perfectly dissolve hydrocarbons at its supercritical state [9], therefore, an additional organic solvent sometimes was used to modify SFE efficiency of target chemicals [28,29].

Several mathematical models (empirical and kinetic models) have been developed to simulate SFE process for various natural products [30–32]. To approximate the experimental extraction data, extraction kinetics is mainly determined by factors including phase equilibrium, mass transfer and solvent flows [32]. Goto et al. provided a quasi-steady state shrinking core model based on the equilibrium of solute–matrix interaction and simulated a short extraction bed without

mass transfer resistance [33]. Reverchon applied the differential mass balances along the packed-bed extraction cell with the heat transfer analogy of a single sphere cooled in the fluid [34]. Sovová proposed a model from the concept of broken and intact cells, assuming that the internal mass transfer from intact to broken cells dominates the rate of extraction [32,35].

The present work describes a study on supercritical CO₂ extraction of solanesol from industrial tobacco waste at various extraction temperatures, pressures particle sizes and CO₂ flow rates. The assistance of organic solvent pretreatment was also employed to improve the solanesol selectivity. The extraction yield and the concentration of solanesol in extracts were analyzed quantitatively. A mathematical model was used to correlate the experimental data. This study provides a new route for generating solanesol via tobacco SFE.

2. Experimental and modeling

2.1. Samples and materials

Tobacco waste (shredded leaves) was from the production line of Shanghai Tobacco Group Co., Ltd. *n*-Hexane (HPLC grade) and methanol (HPLC grade) were obtained from RCI Labscan (Samutsakorn, Thailand). Ethanol (95% purity) was purchased from Yonhua Chemical Technology Co., Ltd. (Jiangsu, China). NaOH (99.9% purity) and HCl (36%–38% concentration) were from Lingfeng Chemical Reagent Co., Ltd. (Shanghai, China). Standard compound of Solanesol (90% purity) was purchased from Sigma Aldrich (Steinheim, Germany). CO₂ (99.999% purity) was supplied by Tianhai Gas Co., Ltd. (Shanghai, China). Synthetic air (20% O₂ + Bal N₂) was from Linde (Shanghai, China). All chemicals were used without any treatment. The mass measurement was conducted on a Mettler Toledo balance with a precision of 0.0001 g.

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