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# Extraction of nicotine from local tobacco using double-supported liquid membranes technique

Zhifeng Guo[a,*], Lei Wang[a,b], Caiyu Song[a], Lixia Zhang[a]

[a] Key Laboratory of Medical Chemistry and Molecular Diagnosis, College of Chemistry and Environmental Science, Hebei University, Baoding, 071002, China
[b] Cangzhou Medical College, Cangzhou, 061001, China

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ABSTRACT

The objective of the study is to modify supported liquid membranes (SLMs) by designing a self-made double-supported liquid membrane (DSLMs). SLMs is a traditional extraction system that adopts organic solvent immersed membrane to separate the donor and acceptor phase. However, the system cannot maintain long extraction time due to the fast evaporation of the organic solvent where the easy volatile organic solvent is selected as organic phase. DSLMs system manages to solve the problem by enclosing the organic solvent between two membranes. This system has been successfully applied to the extraction of nicotine from local tobacco. The optical conditions for extraction nicotine were found and the amount of extracted nicotine were analyzed by GC/MS. Results showed that the extracting time could be held for 24 h and the amount of extracted nicotine got to 43%.

## 1. Introduction

Alkaloids are a kind of hetero-ring compounds with nitrogen which broadly present in plants and herbs. Due to their high bioactivities, alkaloids are often applied in drugs. Therefore, extracting alkaloids from bio-matrix has been an efficient way to develop new drugs. However, since the composition of plants and herbs are extremely complex, it is relatively difficult to extract alkaloids directly from biomass. The traditional methods, such as liquid–liquid extraction (LLE), cannot form delamination due to a large amount polysaccharides containing in the biomass, and not to mention the huge consumption of organic solvents. Until the late 1980s, the support liquid membranes (SLMs), a kind of membrane separation technology, were developed, and demonstrated high matrix removal capacity [1]. Their removal capacity of desired analyte from the sample matrix is conceivable that they could be used to achieve extraction, concentration and cleanup for a wide range of species interested including pesticides, pharmaceuticals, disinfection by-products and metals [2–7].

Typical SLMs is comprised by a hydrophobic porous membrane immersed in an organic solvent. The membrane serves simply as a support for the liquid, placed to separate the feeding (donor) and stripping (acceptor) phases to avoid acidic and basic solution contacting with each other [8,9]. The organic solvent is held in the membrane pores by capillary forces which can attract analyte to be soluble in. Some organic compounds like short carbon chain organic acidic or alkaloid are able to transfer from the donor phase into the acceptor phase to get separated in advance [10–13].

In 1986, G. Audunsoon first applied the technology into separating and enriching samples in analytical chemistry field [14]. Later, Jönsson and co-workers [15–20] did several researches on SLMs about the basic principles, effecting parameters, and device compositions. After that, the technology developed significantly in diverse directions, and came up with several different membrane compositions, such as miniaturization of SLMs [21–27], a porous hollow fiber impregnated with an organic liquid [28–34], and a self-supported liquid membrane extraction device using the electric field as a driving force to enhance transferring [35–38]. At present, certain samples can be concentrated and determined on-line with SLMs [11,39–44].

The efficiency of extraction for the SLMs depends on the amount of analyte transferring through the membrane. The transference is controlled by two factors. One is partition constant of the analyte between aqueous and organic phase; the other is analyte of reactions occurred at the interface between organic phases and stripping phase, which determines the fraction of analyte in extractable form. The organic solvent that has higher solubility in analyte should be in favor of it to transfer. However, alkaloids are not such samples due to their low solubility in straight chain alkane which used as the organic solvent in SLMs [20,45]. On the other hand, SLMs cannot adopt chloroform as the organic solvent for its evaporation will give rise to failure in separating the

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* Corresponding author. Tel.: +86 0312 5079386.
*E-mail address:* gzfvg@hbu.edu.cn (Z. Guo).

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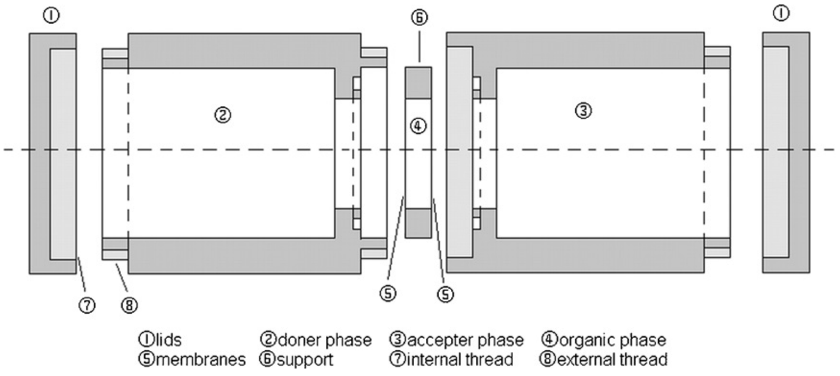

**Fig. 1.** Sketch of DSLM extraction device.

donor and acceptor phase, though alkaloids have higher solubility in it.

Therefore, we designed a DSLMs device with two PVC filtration membranes supported by a nylon hoop to form a room in which the organic solvent can be impregnated. It replaced the single organic immersed organic membrane by two membranes with organic solvent between them. Because the organic solvent can be sealed between two membranes, it assures the organic solvent without fast losing during the extraction. Hence, the chloroform, an easy volatile solvent with high solubility in alkaloids, is able to be used as the organic membrane liquid. As a consequence, the extraction can hold on long time even though the organic solvent is evaporating during the extraction. DLSMs system enhances the extraction, also the stability of membranes.

In this experiment, the efficiency of extraction in the DSLMs device was investigated by using nicotine as alkaloid and trichloromethane as the organic solvent. The extraction efficiency was evaluated by GC/MS. Influence factors, such as the acceptor phase pH, initial concentration of nicotine in donor phase and extracting time were investigated to get the optimal extraction conditions. A local tobacco was treated under these conditions to extract nicotine, through which we also tried to explain the law of transference of nicotine during the DSLMs extraction.

### 1.1. Instruments

The self-made DSLMs extraction device is column aspect (Fig. 1), made by nylon. At the center, an organic room is surrounded by two membranes (0.45 $\mu$m PVC filtration) and a nylon hoop, and is filled with organic solvent during extraction, aims to divide the donor and the accepter phase. And the two phases including lids are connected with screw thread.

The analyses of experiments results were performed by gas chromatography coupled with mass spectrometry. Chromatographic analysis was carried out using an Agilent 6890N GC system coupled with the fused silica capillary column (Hp-5, 32 m, length × 0.32 mm i.d., 1.05 $\mu$m film thickness). Oven temperature was programmed as follows: the initial temperature was 40 °C, and then rose to 140 °C by 40 °C min$^{-1}$, finally, further increased to 220 °C by 10 °C min$^{-1}$, and held for 10 min. The interface line temperature was 220 °C, helium was supplied as the carrier gas in constant flow fashion, the flow rate was 2.4 ml min$^{-1}$, and the column pressure was 35 kPa; each sample (2 $\mu$L) was injected in splitless mode.

For the MS analysis (VG70E-HF, Micromass Corporation) in the total scan mode between 30 and 600$m/z$, an EI source was used, the electron energy was 70 eV, the accelerated voltage was 6 kV, the ion source temperature was 200 °C; And the resolving power was 1000.

## 2. Materials

Tobacco leaves (200 g) were purchased from local market; PVC inert filtration membranes (0.45 $\mu$m pore size) and the chemicals of analytical reagent grade, including camphor, ethanol, trichloromethane, sodium hydroxide, hydrochloric acid and $NaH_2PO_4$ were purchased from Chemical Agent Shop in Baoding, China. A standard sample of nicotine–ethanol (1:1, v/v) was obtained from local Tobacco Co. in Baoding China. The water used in the experiments was doubly distilled water prepared in our lab.

### 2.1. Sample preparation

The alkaloids were extracted by blending 20 g of local tobacco leaves with 200 ml 5% sodium hydroxide for 15 min. After filtered by Brinell funnel under vacuum, the residua were washed by 40 ml distilled water and filter was repeated one more time. The final residua were filtered with absorbent cotton, and washed by a small amount of water. Then, both the filtered and washing liquid were pooled waiting for extraction.

### 2.2. Standard solution preparation

6 $\mu$l nicotine–ethanol solution was pipette into 10 ml volumetric flask, and added to 10 ml with chloroform to make a 300 $\mu$l L$^{-1}$ nicotine stock solution. The stock solution was then diluted to the concentrations of 200 $\mu$l L$^{-1}$, 150 $\mu$l L$^{-1}$, 100 $\mu$l L$^{-1}$, 50 $\mu$l L$^{-1}$ respectively by mixing with trichloromethane by the ratio of 2:1, 1:1, 1:2, 1:5 (v/v). The calibration curve was determined by internal standard method, and camphor was used as internal standard. A piece of camphor was weighed accurately (29.17 mg) and dissolved thoroughly in a small amount of trichloromethane. Then the solution was transferred into a 100 ml volumetric flask, then diluted with trichloromethane to 100 ml to present the internal standard solution. 0.2 ml of the internal standard solution was mixed with nicotine standard solutions and trichloromethane by ratio of 1:2:3 (v/v/v) to present the spiking solution. 2 $\mu$l of the spiked solution was injected into GC/MS and repeated three times. The calibration curve was calculated by using peak area ratios between nicotine and camphor. A linear relationship was obtained with a good correlation coefficient (Fig. 2, $y = 0.4 + 0.0244x$, $R = 0.99732$), when the nicotine solution concentration was as axis $X$, and the ratio of nicotine and camphor chromatographic peak area was as axis $Y$.

### 2.3. Extraction procedure

1.0 ml nicotine–ethanol solution (1:1, v/v) was diluted to 250 ml with distilled water in a 250 ml of volumetric flask to make a 2000 $\mu$l L$^{-1}$ nicotine solution. 15 ml the nicotine solution was