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Application of Non-Newtonian Liquid Membranes to Extract Nicotine from Tobacco

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In the present study, a stable emulsion liquid membrane was prepared through adding polystyrene in a membrane phase. The emulsion liquid membrane extraction system was composed by the membrane phase (kerosene, Span80 and polystyrene) and the strip phase (acidic aqueous solution). The feed phase was nicotine basic aqueous solution or tobacco extractant. A water-in-oil-in-water (abbreviated W/O/W) is formed as dispersing the emulsion liquid membrane with added polystyrene additive in the feed phase, which will be the non-Newtonian. Comparing with the emulsion liquid membranes without polystyrene, the emulsion liquid membranes with polystyrene extended the extraction time from 5 to 8 min, which illustrates that adding polystyrene helps improve the stability of emulsion liquid membranes. The application of using these emulsion liquid membranes for extraction nicotine from tobacco shows the extraction efficiency of nicotine is higher than using liquid-liquid extraction.

Keywords: Emulsion liquid membrane, Polymeric surfactant, Polystyrene, Emulsion stability, Extraction nicotine.

INTRODUCTION

Emulsion liquid membranes (ELMs), also named as multiple emulsions (MEs) in other literature¹ and first discovered Li in the 1960s², is a separation technique that produces an opening of single transmembrane channel in a bilayer lipid membrane. Currently available multiple emulsions consist of water-in-oil-in-water (abbreviated W/O/W) or oil-in-water-in-oil (abbreviated O/W/O) in practical extraction process. A number of applications of emulsion liquid membrane extraction are using W/O/W multiple emulsion system, in which the oil droplets contain fine water droplets and the oil droplets themselves are dispersed in a continuous water phase. The analyte dissolved in the feed aqueous phase (donor phase or external phase) is un-ionized and can permeate from the feed phase (external phase) into the strip aqueous phase (inner phase), where the solute is immediately reacted with the internal reagent converting the un-ionized analytes to an oil-insoluble anion and hence they are incapable of back-diffusion. Formulating an appropriate W/O/W multiple emulsion system, one can easily remove such as acidic compounds³⁻⁷, weak alkali compounds⁸⁻¹² and metal ions from aqueous solution¹³⁻¹⁷. The applications of emulsion liquid membrane also extend to biochemical, biomedical and environmental fields¹⁸⁻²⁶.

According to the literature the aromatic amines can be efficiently removed from wastewater using kerosene and

Span80 as the oil phase and hydrochloric acid as the internal phase. Its removal efficiency achieves 98-99 % within 4 min in a few extracting min^{9,10}. Although emulsion liquid membrane extraction provides the potential for high efficiency separation technique in the clean up sample pretreatments and affecting a diverse number of separation operations, the membrane instability is the major obstacle for its applications at industrial scale.

In general, the breakdown of emulsion liquid membrane stability is described through four possible approaches: (i) coalescence of the internal aqueous droplets into larger internal droplets, (ii) coalescence of the emulsion globules suspended in the external continuous phase, (iii) expulsion of the internal droplets following rupture of the thin membrane film during interaction of the internal and external continuous phases and (iv) swelling or contraction due to water permeation through the oil membrane by diffusion in the case of W/O/W emulsions¹¹. The lack of stability will result into two apparent phenomena in membrane separation processes, (i) loss of the volume of the feed phase and the strip phase, (ii) break of pHs gradient in the feed and the strip aqueous solution, leading to extraction fail or loss efficiency separation, which often occur in the case of W/O/W emulsions.

To solve membrane instability, numerous protocols have been proposed²⁷⁻³¹. One of them is the conversion a Newtonian membrane liquid to the non-Newtonian through adding polymeric

additive in membrane phase, which was proposed by Skelland and Meng²⁸. This protocol was considered as one of the ideal remedy because it succeeds of obtaining greatly increased membrane stability without loss in permeability and with marked reduction in swelling.

Almost all the successful applications of emulsion liquid membrane have the same characteristic that is the concentration of the feed phase aqueous solution is very low. In these cases, there are authors stated that emulsion liquid membranes are ideally suited for the treatment wastewater for removing toxic substance at very low level concentration³². But if the feed phase aqueous solution is bio-matrix, its concentration is no longer low, what happen it will be in the non-Newtonian system? It is known that the stability of emulsion liquid membranes not only relates to the membrane phase but also relates to the feed phase aqueous solution such as its concentration and composition. The studies on this field are seldom. In this paper, we used kerosene and Span-80 as membrane phase, in which polystyrene as polymeric additive was added. This membrane phase was combined with strip phase (acidic aqueous solution) for preparing W/O emulsion at high speed stirring. The feed (donor) phases were the nicotine alkaline solution and tobacco extracting solution. When the added polystyrene emulsion was diffused in the continuous aqueous feed phase at magnetic stirring, a W/O/W emulsion liquid membrane extraction system has been assembled, which would be the non-Newtonian form due to the membrane phase added polymeric additive. The stability and transport properties of this system were investigated through transporting nicotine from the feed to the strip. Nicotine extraction efficiency of using emulsion liquid membrane was compared to that of the using liquid-liquid extraction. Nicotine quantitative analysis was conducted by GC/MS using double internal standard method³³.

EXPERIMENTAL

HP6890N Gas Chromatograph (Agilent Technologies), VG70E-HF Mass Spectrometer (Micromass Corporation), chromatographic column, HP-5 Crosslinking capillary column (32 m × 0.32 mm × 1.05 μm), AD-4 automatic balance (Perkin-Elmer), pHs-2C acidimeter (Shanghai Weiye instrument plant), magnetic stirrer, separating funnel.

Camphor, *m*-cresol, methylene dichloride, ethyl acetate and styrene were all analytical grades and obtained from the Kemiou Chemical Company (Tianjin, China). Ethanol (95 %), kerosene and Span80 (sorbitan monooleate) were purchased from a local chemical shop (Xinhua, Baoding). Nicotine ethanol solution (v:v = 1:1) was used as received from the local cigarette manufacturer (Yulan, Baoding, china). Tobacco leaf (locally produced) was purchased from local market. Polystyrene was purchased from Hujin ethylene limited corporation (Panjin, China). Sulfuric acid and hydrochloric acid with different concentration aqueous solution and sodium hydroxide solution (5 %) were prepared by our laboratory; the water used throughout all the experimental process was double distilled water prepared in our laboratory.

General procedure: A certain volume of the membrane phase (kerosene, polystyrene additive and Span80) mixed with the strip phase solution (0.1 M sulfuric acid) at a certain radio

for emulsification using a mechanical stirrer at agitation speed 4000 rpm. Nicotine ethanol solution (received from the local manufacturer, v:v = 1:1) and tobacco leaf extracting solution were used to prepare a synthetic feed phase. The prepared emulsion was dispersed in the feed phase at magnetic stirring. Samples solutions were withdrawn from the extractor at different intervals of time. The emulsion stability was checked varying pH of the strip phase and the feed phase at different interval time after the emulsion was dispersed in the feed phase. A stable emulsion can keep the feed phase at pH > 7 and the strip phase at pH < 7 long time. The concentrations of nicotine in feed phase or in received phase were measured by GC/MS using internal standard method.

Preparation of water-in-oil emulsion: 2 g of polystyrene was dissolved in 27.2 mL styrene and 22.4 mL kerosene as polystyrene additive marked polystyrene additive. 13 mL of the polystyrene additive was mixed with 0.2 mL Span80 and stirred well in a three-necked flask (250 mL), and then, the mixture was added to 10 mL of sulfuric acid solution (0.1 M) for emulsifying under high speed (4000 rpm) stirring for 40 min. The W/O emulsion prepared with the polystyrene additive was emulsion E1. 13 mL of the kerosene was mixed with 0.4 mL Span80 and stirred well in a three-necked flask (250 mL), and then, the mixture was added 10 mL sulfuric acid solution (0.1 M) for emulsifying under high speed (4000 rpm) stirring for 60 min. The W/O emulsion prepared without the polystyrene additive was emulsion E2.

Preparation of nicotine feed phase solution: Two kinds of solutions were used as the feed phase. One was nicotine aqueous solution and the other was tobacco leaf extraction solution. 0.4 mL of nicotine alcohol solution (v:v = 1:1) was moved into a brown glass volumetric flask (200 mL). The flask was filled with water to scribed line, shaken well as the nicotine feed phase aqueous solution (1000 ppm) marked NS. 20 g tobacco leaf was crushed and soaked in 200 mL sodium hydroxide solution (5 %, mass fraction) in a beaker for 15 min and then filtered using Buchner funnel. The residue was washed twice with secondary distilled water. The filter liquors were combined in a brown glass flask volumetric (200 mL) and then added to secondary distilled water to scribed line as the tobacco leaf extraction feed phase solution (TS).

Emulsion extraction: 30 mL of NS (1000 ppm) was added to 10 mL deionized water in a beaker (50 mL) and then adjusted pH to the appropriate value with NaOH aqueous solution (5 %) and hydrochloric acid solution (6 M) as feed phase. The feed phase was slowly added 10 mL W/O emulsion under magnetic stirring for a nicotine extraction. After extracting for few minute, the mixture of W/O/W was removed into a separating funnel for separate emulsion and the feed phase by gravity. The separate W/O emulsion was heated reflux for demulsification with adding 1.5 mL of ethanol (95 %) and a little NaCl. The demulsified of W/O emulsion was poured in a separating funnel for separating the oil and the strip phase by gravity. The separated strip aqueous phases was adjusted pH to > 8 with sodium hydroxide solution (2 M) and then was extracted with dichloromethane (3 × 4 mL). The extracted dichloromethane was combined and stored in a refrigerator (-20 °C) for GC/MS analysis. The concentration of nicotine in the internal aqueous phase was determined by GC/MS using

internal standard method. Each sample was performed in three times and the mean values were presented.

Tobacco emulsion extraction performance was the same as those of the above except for the feed phase solution was 10 mL of TS with adding 30 mL deionized water.

Chromatographic analysis was carried out using GC/MS (7890A Agilent, Micro-mass Corporation). A FFAP capillary column (30 m $\times$ 0.32 mm $\times$ 0.33 μ m Lanzhou China) was used. The injection port temperature was held constant at 240 °C and operated in an entirely splitless mode and the sample size was 1 μ L. Helium was used as the carrier in constant current mode at rapid 2 mL/min. The temperature program started at 32 °C and was held constant for 1 min and then was ramped at 40 °C/min to 140 °C followed by further ramping at 10 °C/min to a final temperature of 220 °C and then a held for 10 min at this temperature. The total desorption time was 0.5 h which was the time to complete the chromatogram.

The mass spectrometer (VG70E-HF, micro-mass Corporation) was operated using only a single stage of mass detection. EI positive ion mode was used for analysis and the temperatures of the ion source was 200 °C. The electron energy was 70 eV and the collect current was 200 μ A. The vacuum of source was 10^{-4} Pa and analyzer was 10^{-6} Pa. The mass spectrometer was scanned with a scan time of 1 s per scan (m/z 33-600) for data acquisition with a resolution of 1000.

Internal standard solution and standard curve: Internal standard method is used to determine concentration of nicotine in the feed phase or in the feed phase solution. Camphor standard solution was prepared by dissolving the 1.0 g of camphor in dichloromethane in a volumetric flask (200 mL). The concentration of camphor standard solution was 5 mg/mL. *m*-Cresol standard solution was prepared by dissolving 0.2 mL of *m*-cresol in 1.8 mL dichloromethane in a glass test tube. The concentration of *m*-cresol standard solution was 0.1 mL/mL.

A series of concentration nicotine solution (0.12, 0.18, 0.24, 0.3 and 0.36 μ L/mL,) were prepared by diluting a aqueous solution of nicotine (600 μ L/L). Each of them was spiked with 10 μ L of the standard solution of camphor and 1 μ L of the standard solution of *m*-cresol and mixed well as a series of nicotine double internal standard solution. 1 mL of each solution was sampled for GC/MS analysis and 2 μ L of them was injected. Each test was repeated three times. A calibration curve was plotted by measuring five concentrations of the

nicotine double internal standard solutions. The concentration for nicotine was on the X axis and the ratio of nicotine and camphor chromatographic peak areas was on the Y axis. Concentrations of the nicotine solution change plotted against the ratios of nicotine and camphor chromatographic peak areas at different measuring showed a linear relationship with a good correlation coefficient ($Y = 0.007788X + 0.076$, $R^2 = 0.9978$).

RESULTS AND DISCUSSION

Stability of W/O/W: This part should be Extracting of weak basic substance using W/O/W emulsion, the feed phase must be in basic condition and the strip phase must maintain acidic conditions to perform the mass transfer at one direction³. If the basic condition has not been met, the extraction would fail. Table-1 shows the extraction data of nicotine using emulsion E1 (kerosene, Span80 and polystyrene) and emulsion E2 (kerosene and Span 80). From Table-1, the feed phase turned from basic to acid after 5 min of extraction time, which broke the system's mass transfer at one direction. While the feed phase maintained basic after 8 min of extraction when the emulsion E1 was used (Table-1), it illustrates that polystyrene played an important role in emulsion stability. This is probably due to more viscoelasticity in the membrane phase because of the polystyrene being added. The membrane phase contained high molecular weight polymers possess the requisite viscoelasticity due to the polymeric additive long chains. The requisite viscoelasticity may be the key to reduce the swelling and coalescence of W/O emulsion, hence making emulsion more stable.

Another thing worth pointing out is that the pH of the feed phase was decreasing with decreasing the volume of the feed phase using emulsion E2 (kerosene and Span80), but the pH of the feed phase using emulsion E1 (kerosene, Span80 and polystyrene) did not follow a decreasing trend. The pH of feed phase appeared oscillate with extraction time but still maintained basic feed phase. This oscillate are most likely due to the chaos of interface between emulsions and aqueous in the initial extraction. The state of the extraction system extended 3 more minutes, meaning that the emulsion E1 is more stable than emulsion E2. The extended 3 min. was due to the decrease of emulsion swelling and coalescence. The degree of emulsion coalescence and swelling (ECS) can be defined as the ratio of

TABLE-1
CHANGE OF THE VOLUME AND pH OF THE FEED PHASE DURING EMULSION EXTRACTION

Extraction time (min)	The feed phase extracted using emulsion E2 $V_{0e} = 40$ mL $pH_{0e} = 10$ $pH_{0i} = 0.7$				The feed phase extracted using emulsion E1 $V_{0e} = 40$ mL $pH_{0e} = 10$ $pH_{0i} = 0.7$			
	V_{ot}	ΔV	pH_{ot}	ΔpH	V_{ot}	ΔV	pH_{ot}	ΔpH
0	40	0	10	0	40	0	10	0
1	36	-4	8.5	-1.5	38	-2	8.5	-1.5
2	36	-4	8.1	-1.9	38	-2	8.5	-1.5
3	36	-4	7.4	-2.6	41	1	8.2	-1.8
4	36	-4	7.2	-2.8	40	0	7.7	-2.3
5	35	-5	7	-3	40	0	7.5	-2.5
6	35	-5	<6	>3	40	0	7.3	-2.7
7	35	-5	<6	>3	40	0	7.1	-2.9
8	35	-5	<6	>3	40	0	7.05	-2.95

V_{0e} -Volume of the feed phase; pH_{0e} -pH of the strip phase; V_{ot} -Volume of the feed phase after extracting t min; pH_{0i} -pH of the feed phase after extracting t min; ΔV -Change of volume the feed phase after extracting t min; ΔpH -Change of the pH value the feed phase after extracting t min

the volume increment of feed phase at a sampling time (V_{fi}) to the volume of initial feed phase (V_{f0}) and is expressed by

$$\text{ECS (\%)} = \frac{V_{f0} - V_{fi}}{V_{f0}} \times 100$$

Emulsion coalescence and swelling (ECS) is a very important character in emulsion stability. The lower the emulsion coalescence and swelling is, the more stable the emulsion is. Therefore, in Table-1, the volume difference of the feed phase using emulsion E2 is bigger than those of using emulsion E1, which, in another side, also illustrates polystyrene helps improvement the stability of emulsion.

Further study showed that emulsion E1 can be applied to wider pH range strip phase because of adding polystyrene. From Table-2, two different pH (1 and 0.52) solutions were used as the strip phase. The result shows that the feed phase volume difference before and after extraction has not changed or minimal change in Table-2, which may indicate it's more appealing in stronger acid environment. The pH difference before and after extraction has not changed much and the feed phase also maintains basic after 5 min extraction.

Nicotine extraction using W/O/W: Emulsion E1 and E2 was used in nicotine extraction. The feed phase was nicotine aqueous solution. Results are shown in Fig. 1. As we can see, the amount of nicotine extracted using emulsion E1 is significant higher than using emulsion E2. The reason is because the different fluid characteristics. Emulsion E2 is more like Newtonian fluid while emulsion E1 is more like non-Newtonian fluid. The Newtonian fluid has low mass transfer rate among layers which results in lower extraction rate of nicotine. By adding polystyrene, the fluid turns turbulent; this increases the mass transfer rate among layers. Therefore, the nicotine extraction efficiency of emulsion E1 is higher than that of emulsion E2.

Application of emulsion liquid membrane extraction alkaloids from tobacco: We also did a dynamic extraction of alkaloids using tobacco extract water solution as the feed phase. It is found that the majority substance extracted by emulsion liquid membrane is nicotine and the extraction can only hold up to 5 min then the feed phase turned from basic to acid. This was not beyond our expectation, since the tobacco extractant is more complicated than pure nicotine aqueous solution. Even though the emulsion E1 was used the feed phase solution was changed from the basic to the acidic after 5 min extraction. This case was also happened in using E2, in which the basic

Fig. 1. Amount of extraction of nicotine using emulsion liquid membrane with adding polystyrene and without adding polystyrene. The feed phase: 40 mL of nicotine aqueous solution (1000 ppm, pH 10); the emulsion: 10 mL emulsion 1 or 2; the strip phase: 0.1 mol sulfuric acid solution; the agitation speed: 180 rpm

feed phase solution (tobacco extractant) was maintained less 1 min. In order to performance the dynamic test, the pH of the feed phase (tobacco extractant solution) was adjusted to 11. The extracting time can extend to 6 min when the feed phase (pH = 11) was used (Fig. 2). It showed that the amount of nicotine extracted remains constant after 6 min extraction. It mostly likely indicates the nicotine contained in the feed phase is fully extracted by E1. The amount of nicotine transferred is exponential rise with extraction time and rapidly reaches the maximum after 5 min extraction. The same phenomenon has also been found when aqueous solution of nicotine was used as the feed phase (Fig. 1).

Effect of the pH of the feed phase on nicotine extraction: The effect of pH of the feed phase on nicotine extraction was investigated using nicotine extraction solution as the feed phase at four different pH (pH = 8, 9, 10, 11). Extraction time was 1 min. Fig. 3 shows the extraction results. It can be seen that it is a linear relationship between pH and the amount of nicotine extracted (Fig. 2). The amount of nicotine extracted increases about 0.7 μL with pH of the feed phase adding one unit value toward the basic. It indicated the mass transfer rate was favorable to the more basic solution as feed phase.

Effect of adding membrane agent on emulsion extraction: Adding membrane agent can be improved the solubility of analyte in the organic membrane phase, thus, beneficial for the mass transfer from feed phase to strip phase. Chloroform was chosen as the membrane agent adding to the organic membrane phase, acting as a good solvent for nicotine. Considering the density of chloroform is relatively more, adding

TABLE-2
CHANGE OF THE FEED PHASE IN VOLUME AND pH

Extraction time (min)	Feed phase extracted using emulsion E1 in two different pH of the strip solution I and II							
	I (pH = 1)				II (pH = 0.52)			
	V_{ot}	ΔV	pH_{ot}	ΔpH	V_{ot}	ΔV	pH_{ot}	ΔpH
0	40	0	10	0	40	0	8.9	-1.1
1	40	0	9.2	-0.8	40	0	8.9	-1.1
2	39	-1	9.3	-0.7	40	0	8.8	-1.2
3	40	0	9	-1	40	0	8.1	-1.9
4	41	1	8.8	-1.2	40	0	8	-2.0
5	40	0	8.6	-1.4	40	0	7.4	-2.6

V_{ot} -Volume of the feed phase after extracting t min; pH_{ot} - pH of the feed phase after extracting t min; ΔV -Change of volume of the feed phase after extracting t min; ΔpH -Change of pH of the feed phase after extracting t min

Fig. 2. Curve of dynamic extraction of nicotine by emulsion liquid membrane; Feed phase: 40 mL of tobacco extract aqueous solution (pH 11); the emulsion: 10 mL E1; the strip phase: 0.1 M sulfuric acid solution; the agitation speed: 180 rpm

Fig. 3. Influence of the pH of the feed phase on the emulsion liquid membrane extraction; Feed phase: 40 mL of tobacco extract aqueous solution with different pH; the emulsion: 10 mL E1; the strip phase: 0.1 M sulfuric acid solution; the agitation speed: 180 rpm; extracting time 1 min

too much chloroform may lead to the instability of the emulsion due to density changes. 10 μ L chloroform was added in the test. Fig. 4 showed the results of the test. Column 2 was the amount of nicotine transferred using added chloroform emulsion and column 1 was not. Compare to the column 1, the column 2 was significant higher. It proves that higher solubility of nicotine in the organic phase resulted in higher mass transfer rate between the membranes.

Effect of increase the concentration of surfactant in the membrane phase: Surfactants can reduce interfacial tension between the organic phase and the aqueous phase and hence increase emulsion globules, enhance stability^{34,35}. They also increase the total internal interface and the mass-transfer rate during extraction³⁶. On the contrary, surfactants decrease the rate of interfacial chemical reactions leading to reduction in solute transfer rate³⁷. These contrary effects would be happened during extraction but the substantial reduction in diffusivity and permeability would be avoided if the membrane was a non-Newtonian³⁸.

Fig. 4. Influence of the solubility of extract in the membrane phase on the emulsion liquid membrane extraction; A: the polystyrene additive membrane phase was not added chloroform; B: the polystyrene additive membrane phase was added with 0.01 mL of chloroform. 10 mL E1 dispersed in 40 mL of tobacco extract aqueous solution with (pH 11); the strip phase: 0.1 M sulfuric acid solution; the agitation speed: 180 rpm; extracting time 1 min

To test the effect of surfactant on nicotine extraction, 3 different added amount of Span80 (0.1, 0.2 and 0.3 mL) in the membrane phase (13 mL of polystyrene additive with 0.01 mL of chloroform) were investigated. The extraction time was 1 min. Fig. 5 is the nicotine extraction results. It shows that the amount of nicotine extracted is not in linear relationship with the amount of Span80 added in. The amount of nicotine extracted is around 1.8 μ L when the amount added was 0.1 mL. Then it increases to almost 6 μ L when increasing the Span80 to 0.2 mL. While, the amount of nicotine extracted seems to reach constant. It remains around 6 μ L after adding another more 0.1 mL of Span80. It indicates that the improvement on nicotine extraction is limited through increasing the concentration of surfactant in the membrane phase because increased internal interface have been inhibited by an adsorptive barrier which was set up by surfactant.

Fig. 5. Influence of Span 80 in the membrane phase on the emulsion liquid membrane extraction; the amount of the Span 80 added in the membrane phase (13 mL of polystyrene additive mixed with 0.01 mL of chloroform) were 0.1, 0.2 and 0.3 mL, respectively; the feed phase: 40 mL of tobacco extract aqueous solution with (pH 11); the strip phase: 0.1 M sulfuric acid solution; the agitation speed: 180 rpm; extracting time 1 min

Based on all above research, the optimal conditions for nicotine extraction using emulsion liquid membrane are 13 mL of the membrane phase (polystyrene additive mixed with 0.2 mL Span 80 and 10 μ L chloroform) was mixed with 7 mL the strip phase (0.1 mol sulfuric acid solution) under high speed stirring for preparation W/O emulsion. 10 mL of this W/O emulsion dispersed in 40 mL of the feed phase (tobacco extract) for 5 min extraction. The amount of nicotine extracted using emulsion liquid membrane under this optimal condition has been compared with that using liquid-liquid extraction. Results

showed (not listed) that the recovery was 95.1 % and the emulsion liquid membrane extracted nicotine from the same sample are 1.5 times higher than those of extracted by liquid-liquid extraction, which illustrate the extraction of nicotine using emulsion liquid membrane is more efficient.

Conclusion

Polystyrene can be as polymeric additive to be added in kerosene and Span80 for preparation W/O emulsion. The polystyrene helps improvement the stability of emulsion. The emulsion nicotine extraction experiments using this W/O show that (1) extraction efficiency is higher than that of using W/O emulsion without polymeric additive; (2) by adding chloroform in the emulsion, it can largely improve the amount of nicotine extracted efficiency; (3) by adding too much Span80, it hardly improves the nicotine mass flux. When comparing with liquid-liquid extraction, emulsion liquid membrane under optimal extraction condition shows better extraction efficiency. The amount of nicotine extracted using emulsion liquid membrane is 1.5 times higher than those extracted by liquid-liquid extraction.

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