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# SEPARATION OF ALKALOIDS FROM PLANTS BY BULK LIQUID MEMBRANE TECHNIQUE USING ROTATING DISCS CONTACTOR

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**ABSTRACT: -** This paper describes the transport of Alkaloids through Rotating Discs Contactor (RDC) using n-decane as a liquid membrane. The transport of Pelletierine Alkaloid from a source phase through bulk liquid membrane to the receiving phase has been investigated. The general behaviour of Pertraction process indicates that %Extraction of pelletierine Alkaloid increased with increase in the number of stages and the agitation speed but high agitation speed was not favoured due to the increased risk of droplet formation during the operation. The pH of source and receiving phases were also investigated. The effect of organic solvent membrane on the extraction of Pelletierine was evaluated using n-decane, n-hexane and methyl cyclohexane. The results showed that n-decane has a good extracting ability. The highest %Extraction of pelletierine Alkaloid was observed of (69.16%).

**Keywords**: Liquid membrane, Extraction, Pelletierine, Alkaloids

## 1- INTRODUCTION

Liquid membranes have been used extensively in the extraction of both organic molecules such as pesticides and herbicides in waters and wastewater and metals in water and wastewater matrices as well as other organic pollutants [1-2].The using of liquid membranes presents an attractive approach to produce valuable products of high quality and low costs. The using of liquid membranes in the separation processes increases the selectivity and reduce the powerful, toxic and solvents costs than in the case of classical solvent extraction [3-9].

Membrane-based extraction methods have been widely used in the extraction of both ionisable and non-ionisable molecules from different samples [10]. The main attractive features for these techniques are the use of minimal organic solvents, high selectivity and clean-up efficiency, with high enrichment factors. In most cases the overall cost involved is low due to the simplicity of the techniques which normally involve relatively fewer steps and handling procedures as compared to the other sample-preparation techniques [1, 11].

A bulk liquid membrane process for recovery of medicinal compounds from dilute ammoniacal leach solutions was used in this work. Applying Pertraction in RFC, the alkaloid was successfully recovered from model solution of pelletierine, as well as from native aqueous extracts obtained from the roots bark of *punica granatum* L.

Alkaloids, widely existing in natural plants, are compounds containing basic nitrogen atoms. Most of alkaloids are pharmacologically active ingredients in many medicinal plants due to their significant physiological activity. Many alkaloids can be extracted from natural plant materials and purified by modern separation techniques [12]. Pelletierine $C_8H_{15}NO$ is a liquid alkaloid obtained from the root bark of *punica granatum* Linn [13, 11].

The aim of this work is to study the process of pelletierine recovery from its solution using N-Decane as a liquid membrane with a Rotating Discs Contactor technique and apply this technique for selective recovery of alkaloids from native aqueous extraction of Punica granatum Linn roots to produce pelletierine sulphate medicine.

# 2- EXPERIMENTAL WORK:

## 2.1. Reagent and Analytical Methods Used

Various reagents such as n-decane (99% BDH), n-hexane (95% ALDRICH) and methyl cyclohexane (95% HOPKIN & WILLIAMS) were used as a liquid membrane. Ammonia (25% CHEMSUPPLY) and sulfuric acid (98% GCC) were used to adjust the acidity of the aqueous solutions. The concentration of pelletierine in the strip solution was measured by UV-spectrophotometer SP-3000 (OPTIMA INC) at wave length $\lambda$=254 nm. The pH value of the aqueous solutions was measured by means of a laboratory pH meter (CRISON, MM40).

## 2.2. Experimental Equipment

The transport of Pelletierine Pertraction were carried out in a laboratory rotating disc contactor (RDC) made from Perspex (poly methyl methacrylate) as shown in Fig.1. The lower part of contactor is divided, into four compartments: two for the feed and other two for the acceptor solution while the compartments containing the same aqueous solution are interconnected. On the other hand the organic membrane liquid occupies the common upper part of the contactor [14].

Four discs, 1 mm thick and 18 cm in diameter, mounted vertically on a common shaft rotated in each compartment, to provide continuous renewal of the aqueous films and covering the discs as well as the stirring of all three liquids. The lower part of each disc is immersed in the corresponding aqueous solution while the larger upper part is immersed in the organic liquid membrane, as shown in Fig.1.The two stages connected in a way permitting co- counter or batch operation modes. Two peristaltic pumps were used to homogenize the aqueous solutions and to provide samples from each solution. For the Pertraction transport, the following three-liquid-phase system was used:

- Feed (donor) solution (F): 250 ml aqueous solution of ammonia containing 1.13 mmol/litter of Pelletierine C8H15NO;
- Membrane solution (M): 500 ml: n-decane, n-nonane, n-hexane and methyl cyclohexane;
- Stripping solution (S): 250 ml aqueous solution of sulfuric acid.
- For constant shaft rotating in small rpms:
  1- DC-motor (50 rpm),
  2- Variable DC power supply (Smart power system, EMA series).
- During the experimental period the concentration of Pelletierine was measured using an UV-VIS spectrophotometer SP-3000 (OPTIMA INC). The concentration was, also, measured using HPLC and values were consistent with UV values as shown in Fig.2.
- The acidity (pH) of the acceptor and donor solution was measured using pH meter (CRISON, MM40).

## 2.3. Experimental Procedure

### 2.3.1. Leaching

In the beginning five gm. of *Punicagranatum* L. roots were milled to fine powder and leached by 250 ml of buffer solution of ($NH_3$-$(NH_4)_2SO_4$) adjusted to appropriate pH. This solution was shaken for half an hour and filtered to obtain the feed solution.

### 2.3.2. Extraction

The solution from the above step was poured into the RFC using glass funnel. The acceptor solution was adjusted to appropriate pH using few drops of sulfuric acid and placed into the second compartment while remainder volume was filled with the liquid membrane.

### 2.3.3. Separation of Alkaloid in pomegranate root by HPLC

The analysis of the chemical composition was made by High performance liquid chromatography (HPLC). HPLC consists from a mobile phase which is polar and consists of a mixture of solvents such as water and acetonitrile, while the stationary phase comprises of a column which is usually stainless steel and packed with silica particles. A sample of 50µl was injected into the mobile phase using a procedure outlined by Hartley and Buchan and it passes along the stationary phase. The time taken for a sample to pass through the system is recorded which represent the retention time and is considered as one of the characteristic used to identify the compound. All the compounds were separated and identified using HPLC with separation conditions Lichrospher C18 , 3µm, particle size, 50× 4.6 mm internal diameter of the column, detection U.V. set at 254 nm, flow rate 1.2 ml/min. and 30 C˚ temperature, acetonitrile: 0.01M sodium dodecyl sulphate (60:40, V/V).

The sequences of the eluted material to the standard were as follows, in which each standard was 25µg/ml.The area under a peak was used for calculating the concentration of a sample as shown in the following formula below:

$$\text{Conc. of sample } (\mu g/ml) = \frac{\text{Area of the sample}}{\text{Area of the standard}} \times \text{Standard Conc.} \times \text{Dilution factor}$$

The results of the phytochemical screening revealed that (HPLC) shows the relative concentrations ofvarious compounds getting eluted as a function of retention time (Figure 2). The heights of thepeak indicate the relative concentrations of the components present in the plant (Figure 2.a). Results of HPLC referred to the presence of alkaloids Pelletierine only (Figure 2).

## 3-RESULTS AND DISCUSSION:

### 3.1. Choice of Organic Liquid Membrane

The effect of organic solvent membrane on the extraction of Pelletierine was evaluated by using N-Decane, N-Hexane and Methyl Cyclohexane. The results showed that the extraction efficiency of the organic solvent was in the following order: n-decane> methyl cyclohexane> n-hexane as shown thatin Figure.3. Figure 3showed that n-decane gave a good extracting ability and highest Concentration of pelletierine. The increase in molecular weight of the organic compounds resulted in increasing of its organic properties [15]. Since pelletierine is an organic compounds thus its solubility increases with the increase of the molecular weight of the solvent (likes dissolve like). The lowest extracting ability is that of hexane [16].

### 3.2. The Effect of Discs Rotation Speed on Pelletierine Extraction

Pertraction efficiency grows with the increase of disc rotation velocity as shown in Figure 4.This is due to the better agitation of all three phases gives continuous motion of three liquids which provides an intense solute transfer since all consecutive steps of mass transfer proceed by mechanism of eddy diffusion, also the faster renewal of the aqueous films. These results are in agreement with the results obtained by K. Dimitrov et al (2005) [17]. On the other hand the higher rotation speed (higher than 10 rpm) is gives smaller amount of pelletierine sulphate. Higher rotation speed was not favored due to the increasing the risk of droplet formation and process deterioration. These results agree with the results obtained by K. Dimitrov et al [3, 17 – 18]and K. Abed (2014) [19].The variation of pertraction efficiency with the agitation indicates a diffusion control of the process [20].

### 3.3. The Effect of Number of Stages on Pelletierine Extraction

Increasing number of stages in RFC design play an important role to increase the yield or extraction efficiency. The extraction of Pelletierine was carried out using one and two stages andthe results in Figure 5.The using of tow stagesmean four hydrophilic disks which lead to increase in the surface area for contacting with feed solution. Therefore, pelletierine exhausting from roots increase and the same increasing occur in membrane [16]. On the other hand, the increasing in number of stages increases the surface area which is in contact with the acceptor solution .Thus increasing $H_2SO_4$ molecules in membrane. This results in higher conversion than that from one stage [19].

### 3.4. The Effect of the pH in Source phase

The effect of PH in source phase on the concentration of pelletierine transport through liquid membrane was shown in Figure 6. It is quite clear that the transport of pelletierine is influenced by the pH of source phase. The results revealed that the best pH for the source solution gave good extraction of 9.5.

### 3.5. The Effect of the pH in receiving phase

It is observed that the pH of the aqueous acceptor phase played an important role on the extraction of Pelletierine values [9]. Experimental was carried out at various pH values (1.5-3). The concentration and %Extraction of pelletierine increases with increasing pH and the maximum %Extraction is observed at pH of 2, and then it decreases as shown in Figure 7.On the other hand, the stronger acid gives smaller amount of Pelletierine sulphate.

## 4- CONCLUSION:

The Pertraction in rotating film contactor is a suitable technique for pelletierine extraction from its solutions. The results of pelletierine pertraction showed that the n-decane was most suitable for pelletierine recovery. It can be concluded that the best pH of source phase was 9.5 and for receiving phase equal 2 for the extraction of pelletierine; increasing the pH difference resulted in a decrease in final source phase concentration. Increasing the number of stages leads to increase the Pelletierine extraction. Three agitation speeds were used: 5, 10 and 15 rpm. The best speed was 10 rpm where high rotation speed was not favoured due to the increased the risk of droplet formation during the operation.

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**Table (1):** HPLC analysis for standard Pelletierine.

| Seq. | Subjects | Retention Time (min) | Area(nm/min) | Concentration (µg/ml) |
|---|---|---|---|---|
| 1. | Pelletierine (punicine) | 2.95 | 36437 | 25 |

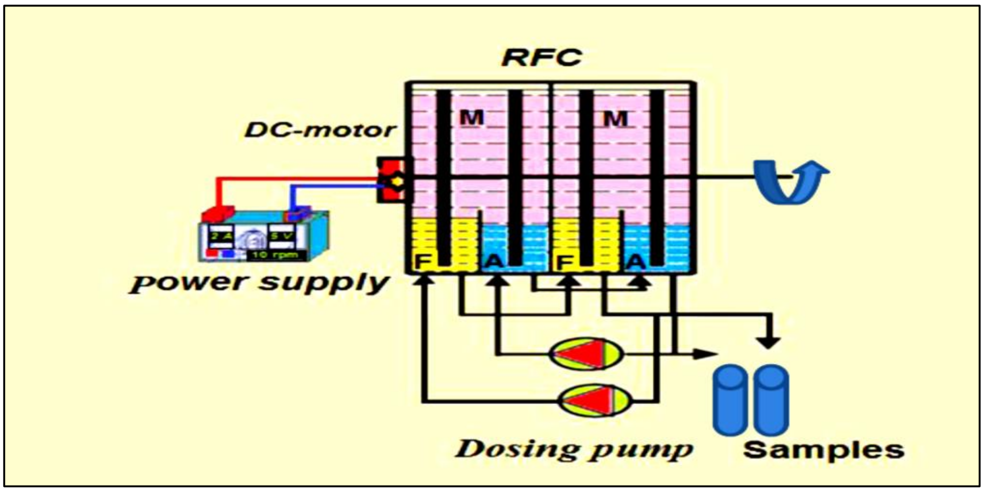


**Fig. (1):** Schematic diagram of rotating disc contactor unit.

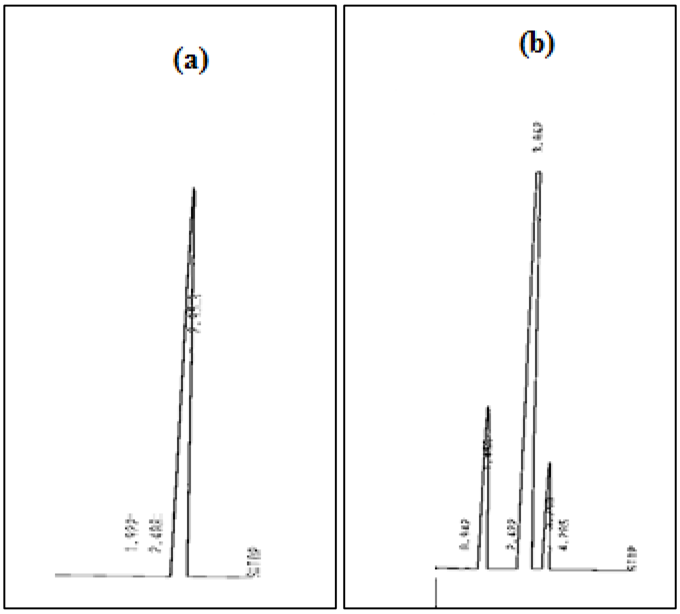


**Fig. (2):** HPLC scanning for (a) Standard Pelletierine (b) Pelletierine extracted by RFC

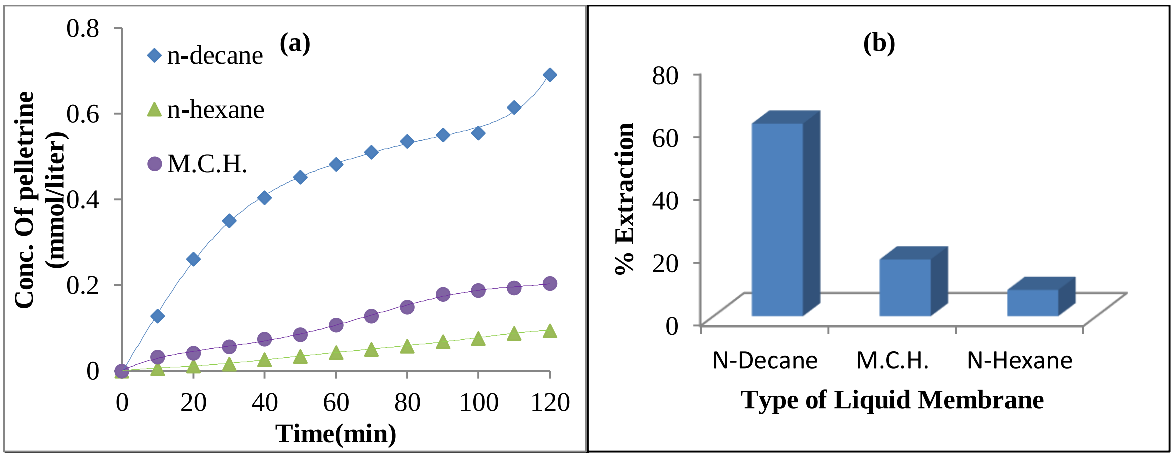


**Fig. (3):** Effect type of membrane on: (a) Pelletierine concentration (b) %Extraction of Pelletierine.

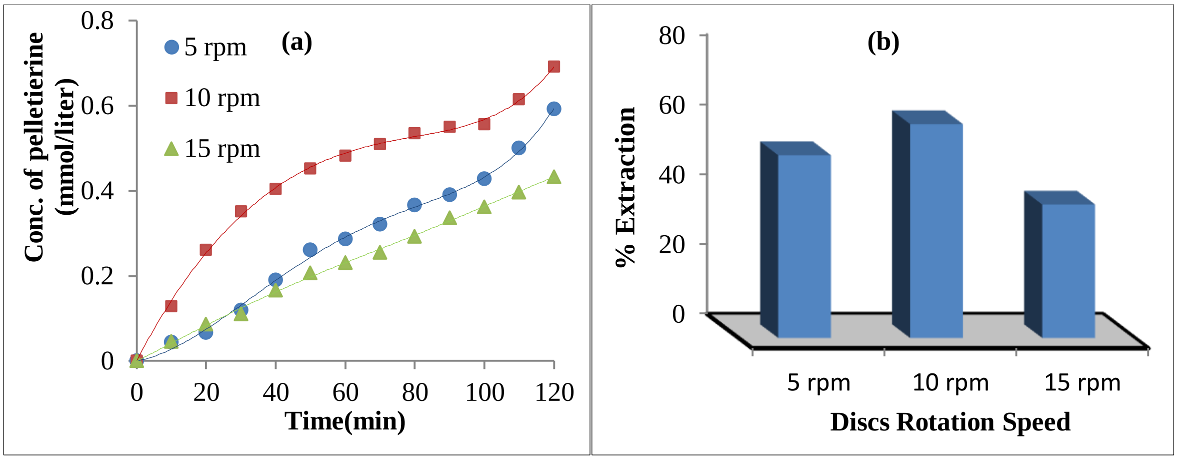


**Fig. (4):** Effect of discs speed on (a) Pelletierine concentration (b) %Extraction of Pelletierine

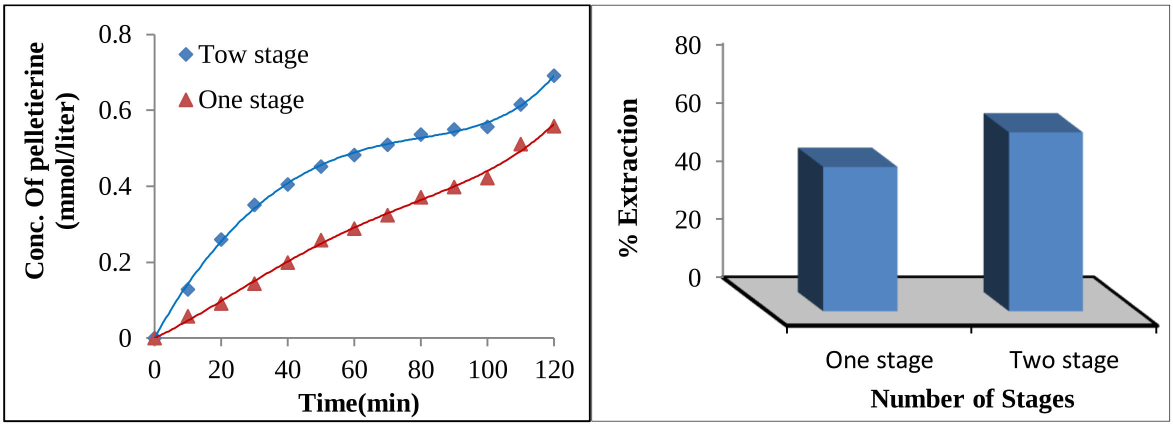


**Fig. (5):** Effect of number of stages on (a) Pelletierine concentration (b) %Extraction of Pelletierine.

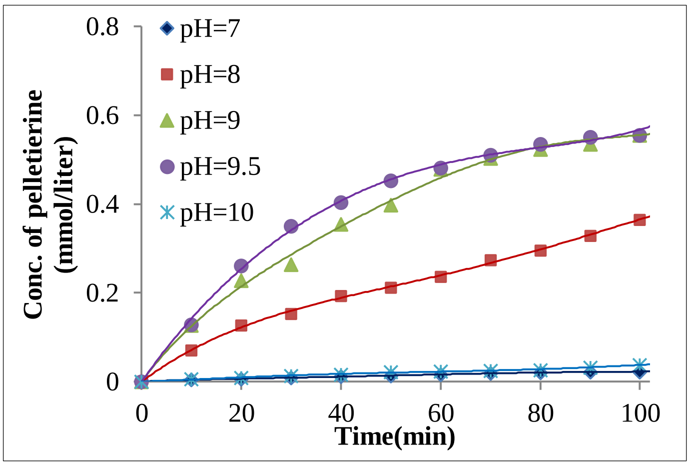


**Fig. (6):** Effect of the pH in Source phase on pelletierine concentration.

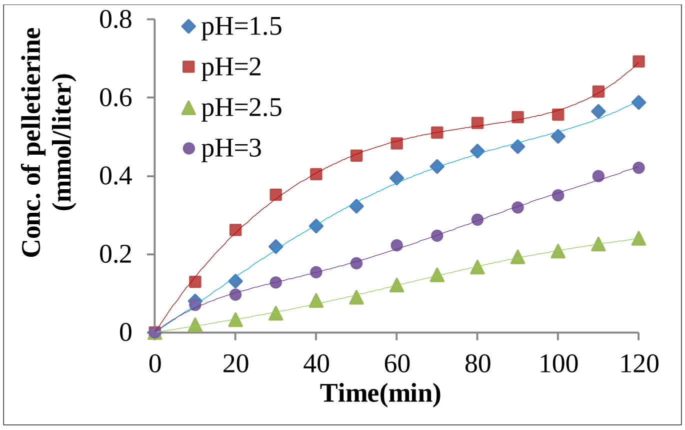


**Fig. (7):** Effect of the pH in receiving phase on pelletierine concentration

# فصل القلويدات من النباتات باستخدام تقانات الأغشية السائلة بواسطة جهاز الأقراص الدوارة

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**الخلاصة**

هذا البحث يصف عملية انتقال القلويدات خلال جهاز الاقراص الدوارة باستخدام الديكان كسائل غشائي. و قد تم تحقيق عملية انتقال مادة البلترين من الطور المصدر خلال السائل الغشائي الى طور المستقبل. ان السلوك العام لعملية الانتقال يشير إلى أن النسبة المئوية لاستخلاص قلويد البلترين تزداد بازدياد عدد المراحل و ازدياد سرعة الخلط لكن سرعة الخلط العالية غير مفضلة بسبب ازدياد خطر تكون القطرات المتكونة خلال العملية. كما تم دراسة تأثير الدالة الحامضية على محلولي الاستلام والمحلول المغذي. تأثير نوع السائل الغشائي العضوي على عملية استخلاص البلترين تم تقييمها ودراستها بأستخدام الديكان و الهكسان و المثيل سايكلوهكسان. اظهرت النتائج ان الديكان له القدرة الجيدة على الاستخلاص حيث كانت اعلى نسبة استخلاص لقلويد البلترين تم ملاحظتها هي 69.16 %.

**كلمات مفتاحية:** الأغشية السائلة، الأستخلاص، القلويدات