IJCPE
Iraqi Journal of Chemical and Petroleum Engineering

Iraqi Journal of Chemical and Petroleum Engineering
Vol.10 No.1 (March2009) 23-27
ISSN: 1997-4884

University of Baghdad
College of Engineering

Extraction of atropine from Datura Innoxia using liquid membrane Technique

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Abstract

Selective recovery of atropine from Datura innoxia seeds was studied. Applying pertraction in a rotating film contactor (RFC) the alkaloid was successfully recovered from native aqueous extracts obtained from the plant seeds. Decane as a liquid membrane and sulfuric acid as a stripping agent were used. Pertraction from native liquid extracts provided also a good atropine refinement, since the most of co-extracted from the plant species remained in the feed or membrane solution. Solid–liquid extraction of atropine from Datura innoxia seeds was coupled with RF-pertraction in order to purify simultaneously the extract obtained from the plant. Applying the integrated process, proposed in this study, a product containing 92.6% atropine was obtained.

Keywords: Extraction, Liquid membrane, Pertraction, Atropine, Purification

Introduction

Atropine sulphate is used widely in medicine for resuscitation, anesthesia, ophthalmology, treat peptic ulcer and as an antidote for poisoning by organophosphate insecticides and nerve gases(1).

Atropine is found in many members of the Solanaceae family. The most commonly found sources are Atropa belladonna, Datura innoxia, D. metel, and D. stramonium. Other sources include members of the Brugmansia and Hyoscyamus genera. The Nicotiana genus (including the tobacco plant, N. tabacum) is also found in the Solanaceae family, but these plants do not contain atropine or other tropane alkaloids.(2)

Atropine can be extracted from the plant as free bases using basic aqueous solutions or as salts using acidified solutions. The obtained aqueous extracts contain many undesirable co-extracted species and the content of alkaloids is rather low. Usually, the obtained native liquid extracts are purified using repeatedly performed solvent extraction operations. The alkaloids are extracted from basic solutions with an appropriate organic solvent. Then, the organic solutions are stripped by acidic solutions and the alkaloids are recovered in the stripping solutions as salts. Because of the relatively low distribution coefficients, for a complete recovery of the alkaloids, both extraction and stripping operations have to be repeated at least three to four times.(4) The difference in pH values between the two aqueous solutions is the driving force in this case.(5)

One of the modern techniques of mixture separation is the application of liquid membranes. They reveal the ability of selective transport of mixture components in which a liquid membrane constitutes a separate phase which separates two other liquid or gas phases. This property of membranes makes them useful in the textile and food industries, in hydrometallurgy, medicine, biotechnology, environmental protection, in the separation of hydrocarbons and gases, and in the concentration and separation of amino acids, metal ions and other mixtures and suspensions.(3) The alkaloid, atropine, is an organic ester which may be prepared synthetically by combining tropine and tropic acid, but is usually obtained by extraction from some Solanaceae plants.(6)

This work was conducted to study the process of atropine recovery from its solution using a liquid

membrane technique and to apply this procedure for selective recovery of the alkaloids from native aqueous extraction of Datura innoxia seeds to produce atropine sulphate.

Experimental

Reagents and analytical methods used

Studies of atropine permeation through the liquid membrane were carried out using atropine aqueous solutions. The atropine was extracted from Datura innoxia seeds (collected in 2004, region of dyala, Iraq) applying solid–liquid extraction. Decane (99%, BDH) as a liquid membrane and sulphuric acid (POCH) as a stripping agent were used. Ammonia solution (CHEM-SUPPLY, 30%), and sulphuric acid were used to adjust the acidity of the aqueous solutions.

Atropine concentration in the aqueous solutions was measured by UV spectroscopy, $\lambda = 257$ nm (λ being the absorbance) and calculated on the basis of atropine (hyoscyamine). For this analysis sulphuric acid and ammonia, as well as diethyl ether (Fluka AG, Buchs SG), heptane (HOPKIN & WILLIAMS), hexane (ALDRICH), Diiso propyl ether (BDH) were used. Since the amount of co-extracted species in the acceptor solutions was found to be insignificant, atropine concentration in these solutions was determined directly by UV-spectroscopy, also the acidity of the aqueous solutions was measured by means of a laboratory pH meter (MAURITUIUS).

Experimental equipment and procedures

The liquid membrane in a rotating film contactor (RFC) offers more intensive hydrodynamics and therefore, a faster pertraction process. Pertraction in a RFC is a special bulk liquid membrane technique in which two aqueous solutions (feed and stripping solutions) form mobile films on the surfaces of vertical rotating discs, partially immersed in the organic membrane liquid. This pertraction technique provides stable and efficient continuous operation, avoiding the phase dispersion or phase intermixing .(7)

The purpose of rotation of hydrophilic discs is to increase the mass transfer effectiveness, the flow of two crossing streams produced in the membrane is in such a way that a vortex flow is achieved. This causes a renewal of the contact area and considerable intensification of mass transfer.(8)

Pertraction studies were carried out in a 3000 ml laboratory rotating film contactor made of Perspex (Plexiglas®) (Fig.1). The apparatus body contained two extraction stages of equal volume. The space in the lower part of each stage is separated into two compartments, for the feed and stripping solutions, respectively. Compartments containing the same aqueous solution are interconnected. The upper part of both compartments contains the membrane liquid. Four discs, 0.5mm thick and 0.17m in diameter, mounted vertically on a common shaft, rotated in each compartment, providing continuous renewal of the aqueous films, covering the discs, as well as the stirring of all three liquids. The lower part of each disc (up to one-third of the disc diameter) is immersed in the corresponding aqueous solution and the larger, upper part is immersed in the organic membrane liquid, as shown in (Fig.2). The discs surfaces being hydrophilic, intended to homogenize the attached aqueous films. Hence, the aqueous solutions occupying the lower parts of each compartment form mobile liquid films on the corresponding disc surfaces, which are in direct contact with the common membrane liquid, filling the upper part of apparatus. The two stages could be connected in a way permitting co-, counter-current or batch operation modes. The latter was chosen in our experiments. To homogenize the aqueous solutions and to provide samples from each solution, both liquids were re-circulated by means of two peristaltic pumps. A variable rotation speed electric motor provided constant shaft rotation.

For the pertraction studies, the following three liquid-phase system was used:

- feed (donor) solution (F): 250 ml aqueous solution
- membrane solution (M): 500 ml
- stripping (acceptor) solution (A): 250 ml

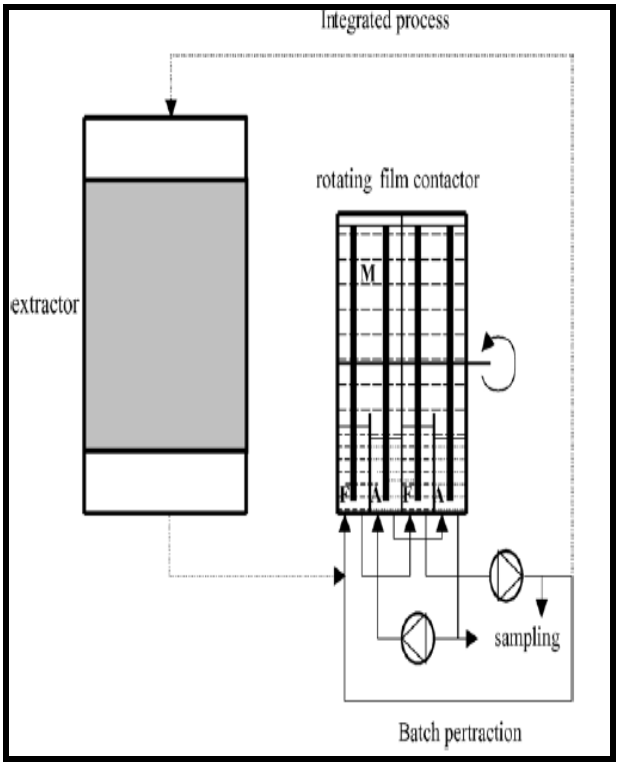

Fig. 1 Scheme of Rotating film contactor [4]

Results and Discussion

The results obtained for the extraction of atropine from Datura innoxia are discussed below.

Effect of type of liquid membrane

Figure (2) presents a comparison of the liquid membranes used. All five LM to some extent show similar behavior.
During experimentation the white crystals of Atropine were observed to start forming after about 15 minutes of operation.
From Fig.(2) it can be seen that decane and heptane gave the highest concentration of Atropine after one hour of operation and then the concentration was steady or dropped only slightly.
However, when using heptane the concentration of atropine decreased, after one hour, because the irreversible reaction lead to atropine transport from acceptor solution to feed solution. Therefore, if heptane is to be used the reaction must be stopped after one hour. As can be seen that the other solvents (hexane, propyl ether and ethyl ether) gave lower concentration values and there was some loss of these solvents during operation due to their high volatility, a problem is not encountered with Decane.
Finally, it should be noted that in the present work decane and heptane were used (for the first time) along with other solvents used by earlier workers (viz; Hexane, Diiso propyl ether and ethyl ether). And Decane was found to be the best solvent for the duty considered, as shown below.

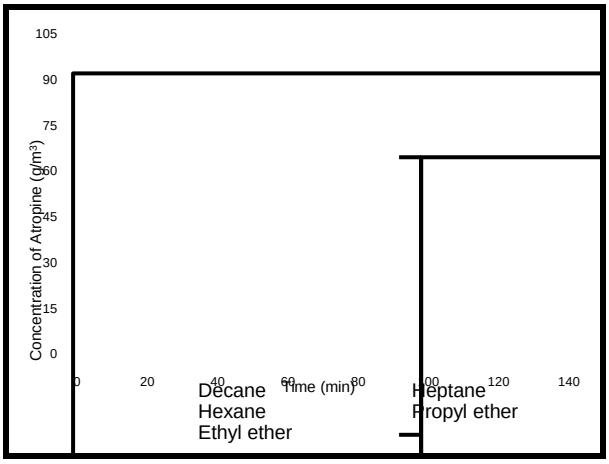

Fig. 2 Effect of liquid membrane used

Effect of pH of the aqueous acceptor solution

From Fig(3), it can be seen that the best results obtained at pH=2.1 . This is because the atropine must be isolated as salt by using dilute acid but if acceptor solution is more diluted which leads to small amount of (H2SO4) molecular in acceptor aqueous solution therefore, being not enough to produce large amount of atropine sulphate. On the other hand when stronger acid is used also gives smaller amount of Atropine sulphate.

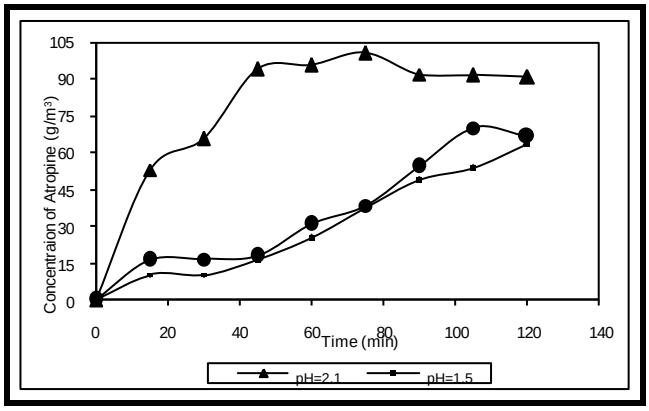

Fig.3 Effect pH of acceptor solution

Effect of amount of seed

When doubling the amount of seeds (7.2g instead of 3.6g) and keeping the conditions constant. This produced higher values of atropine sulphate in comparison with the employed half amount of seeds as shown in Fig (4). Wherewithal these results increased of seeds weight lead to increased amount of atropine in feed solution in the same time in membrane as well as in the stripping phase.
It is reasonable to observe increases in extraction by increasing the atropine concentration in the feed phase. This is true for all extraction processes.

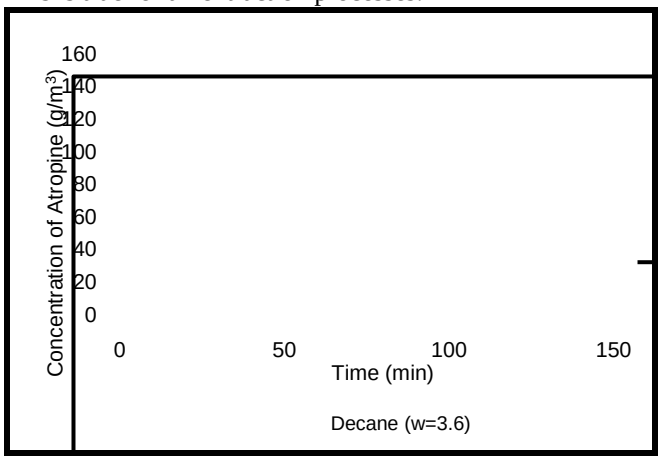

Fig.4 Effect of amount of seeds

Effect of pH of feed solution

It is observed that the pH of the aqueous donor (feed) phase played an important role on the extraction of atropine values. From Fig (5) It can be seen that the best result when pH equal 9.4

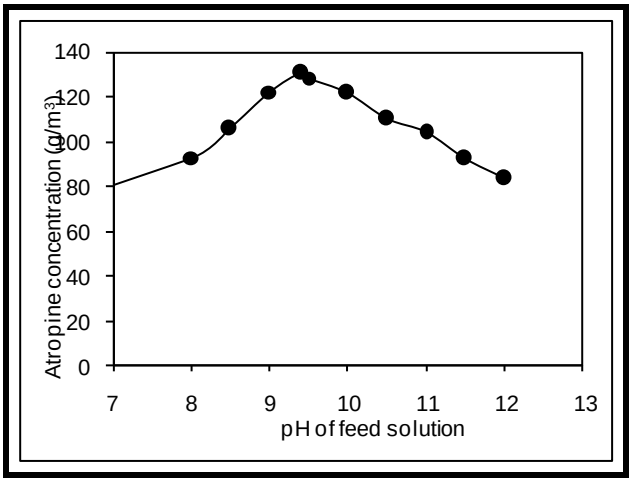

Fig.5, Effect pH of feed solution

In addition, the experiments were carried out under the best conditions obtained, unless otherwise stated, these conditions are: pH of feed solution 9.4, rpm=10, weight of seeds=3.6gm, pH of acceptor solution 2.1 and two stage unit.

Yield

Datura innoxia contain atropine, hyoscine as well as small amount of hyoscyamine and other alkaloids. Generally the content of alkaloids in Datura is (0.01 - 3 %) from dry weight, this percentage depended on plant type, environmental and agriculture process.(9),(10)

From Herbal India Constitution the major chemical compounds found in Datura innoxia is 33% atropine, 66% hyoscine and 1% hyoscyamine oxide and other compounds.(11)

Therefore the maximum concentration of atropine in seeds is theoretically (3% * 0.33 * 3.6gram/ 250cm3) equal o.1425 * 10-3 (gram/ cm3) = (142.5 gram/m3), but from our experiments on the local plant the concentration was found to be (132.54 gram/m3)

And the following table shows the amount of atropine in feed and in product, as well as the yield(12).

solvent	Atropine in feed gram/ m^3	Atropine in product gram/ m^3	Yield %
Decane (two stage)	130.45	120.855	92.645
Decane (one stage)	130.45	100.91	77.35
Heptane	130.45	95.148	72.94
Hexane	130.45	31.42	24.086
Diiso propyl ether	130.45	48.116	36.88
Ethyl ether	130.45	27.292	20.92

Conclusions

Pertraction in a rotating film contactor is a suitable technique for atropine recovery from its solutions, including native liquid extracts of Datura innoxia . The process of atropine recovery from the plant seeds using decane as a liquid membrane is very selective.

1. The highest atropine yield (92.6%) was achieved when using two stages.
2. It was found that the decane is the best solvent as liquid membrane.[12]
3. Best pH value of acceptor solution is 2.1 and of feed solution are 9.4.
4. It was found that the atropine conversion increases with increasing amount of Datura seeds feed.
5. The hydrophilic discs of stainless steel gave good extraction.
6. Increasing the number of stages caused increased atropine extraction.

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