International Food Research Journal 24(Suppl): S540-S543 (December 2017)
Journal homepage: *http://www.ifrj.upm.edu.my*

Characterization, purification and identification of some Alkaloids in *Datura stramonium*

[1]Babiker, F., [*1,2]Jamal, P., [1,2]Mirghani, M.E.S., [2]Ansari, A. H

[1]*Department of Biotechnology Engineering, Kulliyyah of Engineering, International Islamic University Malaysia (IIUM), P. O. Box 10, Gombak, 50728, Kl, Malaysia*
[2]*International Institute for Halal Research and Training (INHART), Kuala Lumpur. Malaysia*

Article history

Received: 7 July 2017
Received in revised form: 8 July 2017
Accepted: 8 July 2017

Keywords

Datura stramonium
Seeds]
TLC
FTIR
Atropine
Hyoscine

Abstract

This study aimed at investigating the presence of alkaloids and other chemical constituents in *Datura stramonium* (Saikaran, Jimson weed). All parts of the plant were dried, crushed and then underwent extraction by soxhlet and maceration methods. The solvents used in these methods were normal hexane (nonpolar) and ethanol (polar). Thin Layer Chromatography (TLC) and FTIR techniques were used to analyse the chemical components of jimson weed. The results showed the presence of hyoscine in all plant parts while atropine in the seeds only. The best separation was found to be when the solvent system was acetone: water: ammonia (90:07:03). Maceration method is the best and cost effective procedure for extraction.

Introduction

The study of natural products has played a major role in the development of organic and medicinal chemistry, including fundamental aspects of stereochemistry, mechanistic chemistry, biosynthesis and mechanism of biological action, as well as providing medically useful compounds.

Amongst natural products, it is the secondary metabolites which give a particular species its characteristic features. Unlike primary metabolites, these compounds are neither ubiquitous in the living organisms that produce them nor are they expressed continuously plants are the best-known sources (Jamal, 2016). Standard separation and analysis methods are commonly used in alkaloids identification, with the hyphenated method as HPLC-MS and GC – MS, having a particular value. Carbon 13 and proton NMR methods are useful in structural elucidation and biosynthetic studies (Cordell, 1981).

Datura stramonium is commonly known as Devil's trumpet, Jamestown or Jimson weed (Lee, 2007). It's family is Solanaceae, the nightshade family and deadly nightshade (Atropabelladenna), are rich in alkaloids. A weed belonging to the family Apiaceae, *C.maculatum* is one of the most common poisonous plants found in the northern hemisphere. It was used as a drug, however, its medicinal importance is now very limited due to the small difference between it's therapeutic and toxic dosage (Vetter, 2004).The principal alkaloids in the plant are the piperidine alkaloids, coniine and γ-coniceine, which are present in ~100 – 1000 $\mu g g^{-1}$ quantities and N-methyl coniine, conhydrine, Pseudoconhydrine, which are secondary alkaloids present in ~10 – 100 – $\mu g g^{-1}$ quantities (López *et al*., 1999). *Datura stramonuim* and *A. belladonna* are rich in tropane alkaloids; primarily atropine and scopolamine, (Friedman and Levin, 1989; Philipov and Berkov, 2002).

Figure 1. Diagram of fresh plant of *Datura stramoonium*

The picture of the *D. stramoonium* is shown in Figure 1. It has been classified that, Jimson weed is the common name and *Datura stramonium* is the scientific name of this plant. It is stated that

*Corresponding author.
Email: *jparveen@iium.edu.my, jparveen3@yahoo.com*

the habitat of this weed is found in cultivated and distributed areas throughout the southern United States, and throughout temperate and tropic areas of the world.

It is also reported that *Datura* is grown in full sun in moisture retentive but well drained fertile and preferably calcareous soil, *Datura* seems to occur naturally on fertile wasteland, rubbish tips, dry river banks and roadsides. They almost always start growing where the ground has been recently disturbed. The main aim of this study is to investigate the presence of alkaloids in the extract of *Datura* stramonium (Jimson weed), using TLC and IR.

Materials and methods

Sample preparation

The fresh sample of *Datura stramonium* used in this study was originally brought from farms near to the University of Gezira in Wad Medani town, Sudan. The samples were cleaned and the stem, seed, leaves and roots were manually separated. All the plant parts were subjected to drying at room temperature for 4 days. The dried samples were blended using electronic blended into powdered and allowed to pass through a sieve of 5µm mesh size.

Extraction

The crude extracts of a different part of *D. stramonium* were obtained using maceration method with ethanol and hexane as the extraction solvents. About 60 mL ethanol was added to each of 5 g leaves, stem, seed, and root in a 250 mL cornical flask. The 250 mL canonical flask covered with a cotton, plastic stock and aluminium foil. Extraction was conducted for six days with occasional shaking. The shaking was done manually at an interval of 12 hrs. The soluble extracts were collected by filtration through 1 mm diameter filter paper under gravity. The extract obtained was clarified and then concentrated. During hexane extraction, a volume of 100 mL n-hexane was added to each of 5g leaves, stem, seed and root. All other steps were similar to that of ethanol described earlier (Djilani *et al*., 2006). The extracts were stored in chiller until needed for analysis.

Determianton of yield of extract

The percentage yield of the extracts was determined using the Equation 1.

%yield=(weight of the extract)/(initial weight of sample) x100 (1)

TLC solvent system

Toluene: ethyl acetate: diethyl amine, (70: 20: 10). Acetone: water: concentration. Ammonia (90: 07: 03).

Thin layer chromatography

Glass plates (20 x 20 cm) had been carefully cleaned with acetone to remove grease, then the slurry of silica gel (F_{254} with $CaSO_4$) in water vigorously shaken for a set time interval, 80 mL water, 40 gm silica, before spreading plates, after spreading air dried then activated by heating in an oven at 100 – 100° for 30 minutes. The choice of the solvent system depends on the properties of the components to be separated. A concentrated solution of the sample and authentic sample were placed at one end of the adsorbent layer, a label from left to right as follows: Hyoscine, atropine, seed, stem, root, leaves, atropine and hyoscine.

Then the plate was placed horizontally into a tank containing the element, so that the sample was little above the level of the solvent. When the solvent front has moved a sufficient distance, the chromatogram was developed. In all cases, the spots in the developed chromatographic plates were located by examination under an ultraviolet lamp. The components can be identified on the basis of the (R_f) values determined from the chromatogram, calculated according to Equation 2.

R_f=(distance of the solute)/(distance of the solvent moves) (2)

Where: R_f = Retention factor.

Infrared spectroscopy

The compound is dissolved to give, typically, a 1-5% solution in carbon tetrachloride or, for its better solvent properties, alcohol free chloroform. This solution is introduced into a special cell, 0.1-1 mm thick, made of sodium chloride. The second cell of equal thickness, but containing a pure solvent, is placed in the path of the other beam of the spectrometer in order that solvent absorptions should be balanced.

Results and discussion

Effect of solvent on the percentage yield of extraction

Figure 2 shows the result of the pecentage yield of crude of *D. stramoonium* obtained from marceration using hexane and ethanol. The percentage yield varies from different parts of the plant and the type of solvent used. The values of the yield of the extract range from 1.25–2.54%, respectively, for hexane extract of seed and ethanolic extract of leaves. The

Table 1. R_f of different fractions of solvent extraction on crude extract from *D. stramonium*

	Ethanolic extract				Hexane extract			
	seed	stem	root	leave	seed	stem	root	leave
Retention factor (R_f)	16	13.4	9.7	7.8	16.4	12.7	5.1	7.3

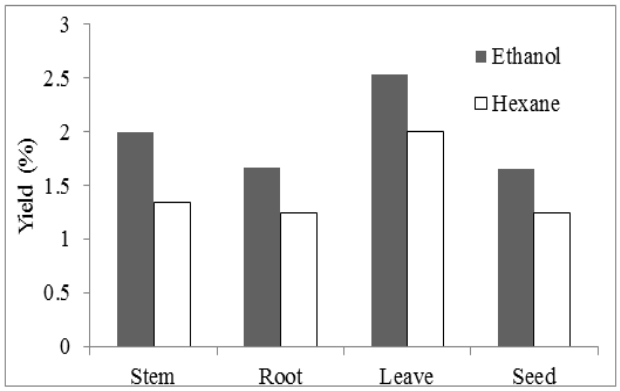

Figure 2. The percentage of ethanolic and hexane extracts of *Datura stramoonium* obtained using merceration method

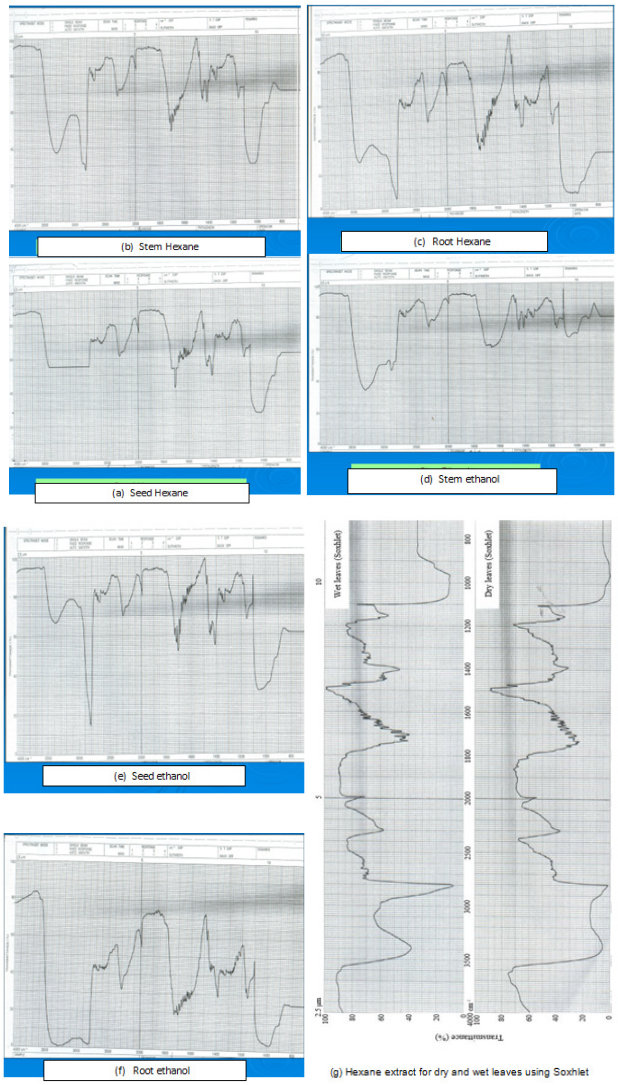

Figure 3. IR spectrum for all plant parts

yield of ethnol from all the samples was higher than the yield of hexane. This suggests that the samples contain more polar biomolecules than non polar ones (Yubin *et al*., 2014). The maximum yield of ~2.5% from the ethanolic extract of leaves was similar to that result of 2.79% obtained from a methanolic extract of *D. metel* (Alabri *et al*., 2014).

Alkaloid compounds separated by TLC

The detected alkaloids by TLC plates using dragendorff reagent insured that all the extracts of *D. Stramoonium* (root, stem, seed and leaves) contain hyoscine, the atropine appear only in the spot of the seeds under the U.V lamp. The ethanolic extraction with the two different solvent systems shows the same behavior at 365 and 354 nm. The same yellow color appears and the chlorophyll reflection in red color; notice that these color of the spots were unstable. Table 1 shows the number of components and their R_f values using different solvents:

Infrared interpretation results and discussion

Each of the spectrums shown in figure 3 which the IR spectrum insured absorption of bands in the region of 3000–3700 cm^{-1} (2.3-3.3 µm) and that explains the presence of OH or NH functional group in the compounds (s) which are under study. Hence, alcohols and amines also exhibit C-O and C-N absorption in the finger–print region from 900-1300 cm^{-1} (8-11 µm), (even it's not easy to identify). Therefore, we can suggest that structure of the compound (s) got these functional groups. Carbon-carbon double bond absorption 1980 - 1620 cm^{-1} aromatic part.

Bonds near (1600, 1580, 1500, 1450 cm^{-1}) are sharp with the variable intensity they due to carbon-carbon bond vibrations of the aromatic ring. The absence of absorption by a compound in this region indicates that the compound is not aromatic. Aromatic C-H bonds 3100-3000 cm^{-1}. C-H bending vibrations 900-650 cm^{-1} are diagnostic of substitutions of the aromatic ring. IR intense peak 1700 cm^{-1} aryl ester 1580 cm^{-1} + aromatic peak- conjugation with benzene ring ~ 2800 cm^{-1}. The absence of strong absorption in the position 2800–3000 cm^{-1} (3.1 – 3.75 µm), that indicated surely that the compound(s) is not an aliphatic. Finally, even these information's were not satisfied to make most clear-cut answer for

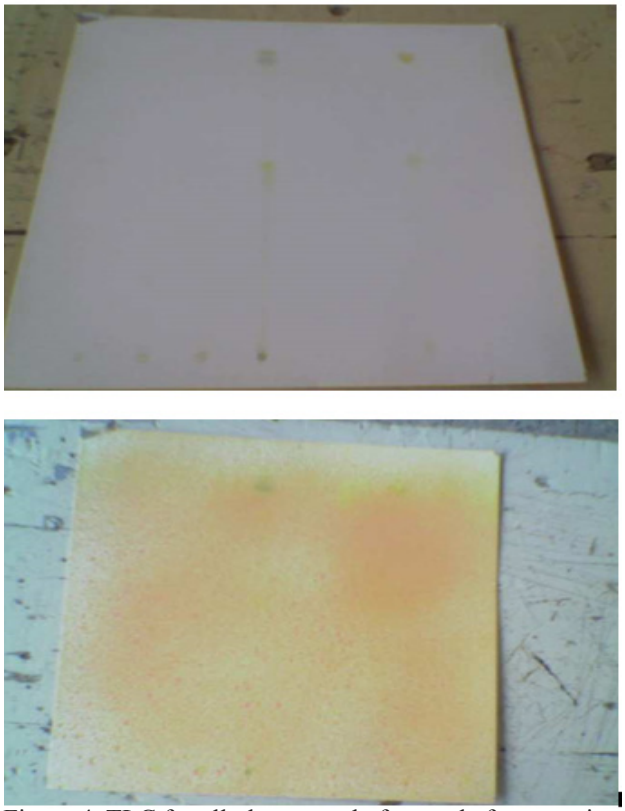

Figure 4. TLC for all plant parts before and after spraying

determining the structure of atropine and hyoscine surely, but we suspect that the compound(s) under study contain(s) these functional groups which atropine and hyoscine and the other related compounds. Therefore, spectroscopy techniques as GC, GC–MS, and NMR, if in hand can be used to give the clearest cut answer on this side.

Conclusion

Maceration method is cheap and it is the best procedure for extraction especially when ethanol is used. The main components of the extraction can be separated by TLC and IR techniques. Furthermore, the experiment shows that there was more yield of ethanol than hexane from all the samples suggesting that the samples contain more polar biomolecules than non polar ones. The maximum yield of ~2.5% from the ethanolic extract of leaves was similar to the result of 2.79% obtained from a methanolic extract of *D. metel* (Alabri *et al*., 2014). The results from infrared spectroscopy also show that alkaloids contain various functional groups such as alcohol and amines.

References

Alabri, T.H.A., Al Musalami, A.H.S., Hossain, M.A., Weli, A.M. and Al-Riyami, Q. 2014. Comparative study of phytochemical screening, antioxidant and antimicrobial capacities of fresh and dry leaves crude plant extracts of *Datura metel* L. Journal of King Saud University - Science 26: 237-243.

Cordell, G. 1981. Introduction to Alkaloids A Biogenetic Approach, 1981. New York: A Wiley Interscience Publication, John Wiley and Sons.

Djilani, A., Legseir, B., Soulimani, R., Dicko, A. and Younos, C. 2006. New Extraction Technique for Alkaloids. Journal of the Brazilian Chemical Society 17: 518-520.

Friedman, M. and Levin, C.E. 1989. Composition of jimson weed *(Datura stramonium)* seeds. Journal of Agricultural and Food Chemistry 37: 998-1005.

Jamal, P., Akbar, I., Hashim, Y.Z.H. and Jaswir, I. 2016. Process development for maximum lycopene production from selected fruit waste and its antioxidant and antiradical activity. Journal of Food Processing and Technology 7(4): 1-7

Lee, M. 2007. Solanaceae IV: Atropa belladonna, deadly nightshade. The Journal of the Royal College of Physicians of Edinburgh 37: 77-84.

López, T.A., Cid, M.S. and Bianchini, M.L. 1999. Biochemistry of hemlock (*Conium maculatum* L.) alkaloids and their acute and chronic toxicity in livestock. A review. Toxicon 37: 841-865.

Philipov, S. and Berkov, S. 2002. GC-MS investigation of tropane alkaloids in *Datura stramonium.* Zeitschrift für Naturforschung C 57: 559-561.

Vetter, R.S. 2004. Myths about spider envenomations and necrotic skin lesions. The Lancet 364: 484-485.

Yubin, J., Miao, Y., Bing, W. and Yao, Z. 2014. The extraction, separation and purification of alkaloids in the naturalmedicine. Journal of Chemical and Pharmaceutical Research 4: 338-345.