Pak. J. Bot., 51(2): 567-572, 2019. DOI: 10.30848/PJB2019-2(9)

EXTRACTION AND BIOACTIVITY FROM *JATROPHA CURCAS* L. LEAVES BY STEAM DISTILLATION

LAURA SOTO-ARMENTA[1], JULIO SACRAMENTO-RIVERO[1], AZUCENA GONZÁLEZ-COLOMA[2], PABLO ACERETO-ESCOFFIÉ[1], ERICK AGUILERA-CAUICH[3], GREGORIO MARTÍNEZ-SEBASTIAN[3] AND J. ANTONIO ROCHA-URIBE[1*]

[1]Universidad Autónoma de Yucatán. Facultad de Ingeniería Química, Periférico Norte km 33.5, Tablaje Catastral 13615, Col. Chuburna de Hidalgo Inn, Mérida, Yucatán 97203, México
[2]Instituto de Ciencias Agrarias, CSIC, Serrano 115-dpdo, Madrid 28006, España
[3]Agroindustria Alternativa del Sureste - Grupo Lodemo, Parque Industrial Yucatán, Yucatán 97300, México
[*]Corresponding author's email: rochaja21@hotmail.com

Abstract

Jatropha curcas L. has biological activities that can contribute to find new products. In this study, steam distillation at laboratory scale was applied to *J. curcas* leaves to assess the yield of essential oil and the bioactivity of the hydrolate. The effect of steam flow and bed porosity on the extraction yield was also determined, where it was observed that residence time was one of the influential factors in the yield of essential oil. The extracts were analyzed by GC-MS. Dibutyl phthalate, phytol, and diisooctyl phthalate were the majoritarian components. Research reports indicate that these components have biological activity. The greatest yield obtained was 0.38% on a dry weight basis. The bioassays showed that the hydrolate of *J. curcas* possessed toxicity against *Spodoptera littoralis*, *Myzus persicae*, *Lactuca sativa*, and *Lolium perenne*. The bioactivity of these products should be further explored, they have a promising future as a biocontroller.

Key words: Steam distillation, Bioassay, *Jatropha curcas*, Yield, Antifeedant, Phytotoxic.

Introduction

Jatropha curcas L., is a multi-purpose shrub, traditionally used as a medicinal plant and currently as a source of vegetable oil for biodiesel. Diverse studies report that the leaves extract present anti-diabetic and anthelmintic properties along with insecticide, antibacterial, and nematicidal activities (Pabón & Hernández, 2012). A phytochemical analysis identified the presence of flavonoids, steroids, saponnins, alkaloids, tannins, triterpenoids, carbohydrates in the leaves, in ethanolic extracts β-stigmasterol and phytol were identified (Ahirrao *et al*., 2011; Ma *et al*., 2011a). β-stigmasterol has acaricidal activity while phytol has antimicrobial, anti-inflammatory, anticancering, and diuretic activities (Rajeswari *et al*., 2012).

Some studies reported that aqueous and methanolic extracts had insecticidal activity specifically against *Anopheles arabiensis*, *Culex quinquefasciatus* and avoid egg hatching from *Rhipicephalus annulatus.* Hexane extracts proved to be a potential biopesticide against *Musca domestica*. Methanolic and ethanolic extracts have allelopathic activity inhibiting the growth of wheat seeds (*Triticum aestivum*) (Kovendan *et al*., 2011; Tomass *et al*., 2011; Juliet *et al*., 2012; Reichel *et al*., 2013; Chauhan *et al*., 2015; Khattak *et al.,* 2015).

Spodoptera littoralis and *Myzus persicae* are important pests for a wide range of crops, vectors of diseases that cause great economic losses. The phytotoxic effect of a certain product can be used as an indicator, in this study seeds of *Lactuca sativa* and *Lolium perenne* were used, these seeds were easily accessible and the results were obtained in short time, showing the effects of the extracts on root length and germination (Martín, 2012).

Essential oils are the final product of the synthesis of plants from their secondary metabolites and are produced in small quantities. Depending on the plant, yields can vary from 0.5 to 6%, and these oils have a wide range of applications in three main sectors: food, pharmaceutical, and cosmetic industries. The functions of these oils are diverse ranging from the attraction of pollinators, defense against herbivores and pathogens, and inhibition of the growth of other plants around them (Stashenko, 2009; Ordaz *et al*., 2011; Calvo, 2012).

During the steam distillation of medicinal and aromatic plants, an aqueous solution known as hydroaltes is obtained, which is composed of many bioactive hydrophilic compounds (Rabha *et al*., 2012).

A high extraction yield and a high concentration of bioactive compounds (with antioxidant, antibacterial, or insecticide activities, etc), are the main characteristics of essential oils which determine their potential value for extraction and commercialization, produced and. (Calvo, 2012). The oils obtained from plants, due to their medicinal properties, low toxicity, and practically null side effects, are increasingly used in the food, pharmaceutical, and agro industries (Salomón *et al*., 2009).

In this study, steam distillation at laboratory scale was applied to *J. curcas* leaves to assess the yield of essential oil and the bioactivity of the extract. The effect of steam flow and bed porosity on the extraction yield was also determined.

Materials and Methods

Plants materials: Fresh leaves from a plantation located on marginal lands in Tizimín (Yucatán, Mexico) were provided by the Company Jatronergy (Lodemo corporate group) *J. curcas* The leaves were taken from the November-to-June pruning season and dried in a vented and roof-covered room at ambient temperature for 3 weeks until reaching a moisture content of about 10%. The leaves were used without separation or ground.

Experimental devices: Steam distillation equipment at laboratory scale experiments were carried out in a 4.5 cm diameter and 0.334 L capacity glass (Fig. 1), located in the Faculty of Chemical Engineering of the Universidad Autónoma de Yucatán (FIQ-UADY). In these experiments, 6.33 g (0.90 bed porosity) and 19.07 g (0.79 bed porosity) of fresh dried *J. curcas* leaves were packed and extracted with steam for 4 hours. This set is similar to one reported for *Jatropha gossypifolia* leaves extraction (Aboaba *et al*., 2015). The extract obtained from the *J. curcas* leaves was collected and the essential oils were extracted from the hydrolate using dichloromethane (Hossain *et al*., 2012). The essential oil was then dried with anhydrous sodium sulphate (Na_2 SO_4) and stored at 4C for further analysis.

Fig. 1. Steam distillation equipment at laboratory scale.

Experimental design: A total of 8 tests were established for *J. curcas* under a 2×4 factorial design (the two factors being bed porosity and steam flow). The bed porosity levels were 0.90 and 0.79, and the steam flow levels were 0.73, 1.78, 4.33 and 8.10 $cm^3 \cdot min^{-1}$. The levels of bed porosity were chosen between loose and tight (Koul *et al*., 2004), and the steam flow were fixed according to the minimum and maximum thermal load. The equipment was operated at atmospheric pressure and the steam flow was obtained by measuring the hydrolate through volumetric analysis (Cerpa *et al*., 2008). The bed porosity (ε) was estimated as $\varepsilon = 1 - d_l/d_s$, where d_l is the apparent density of the bed and d_s is the density of the vegetable matter, determined by helium pycnometer (Kotnik *et al*., 2007; Cerpa *et al*., 2008; Özek, 2012). The essential oil yield (Y) was estimated as $Y(\%) = M/M_0 \times 100$, where M is the mass of essential oil obtained and M_0 is the initial amount of vegetable matter introduced to the process (Cerpa *et al*., 2008; Özek, 2012).

The results of each experimental design were analyzed using a two-way analysis of variance (ANOVA). The statistical significance of the results were assessed with a Tukey test using STATGRAPHICS software.

Essential oil analysis: An Agilent Technologies chromatographer (model 6890N) coupled to an Agilent 5973N mass selective detector (MSD) with an electron impact ionization of 70 eV was used. The injection volume was 2 µL (4% essential oil/CH_2Cl_2 v/v) using a 100:1 split ratio. An HP-5MS (30 m x 250 µm x 0.25 µm) column was used. The oven operating conditions were: initial temperature of 50°C for 3 min, then rising from 50°C to 250°C at 20°C/min and finally kept isothermal at 250°C for 10 min before post run (280°C for 5 min). Helium was used as the carrier gas at 1 mL/min. The injection and transfer line temperature were 150°C and 280°C respectively. The chemical compounds were tentative identified by comparing their mass spectra with those compiled in the NIST 2011 library (Ma *et al*., 2011a; Ma *et al*., 2011 b).

Antifeedant activity: For determining the antifeedant activity, *Spodoptera littoralis* sixth instar larvae were used. Leaf disks of *Capsicum annuum* with an area of 1.0 cm^2 were treated on the upper surface with 10 µL of the test substance. Two *S. littoralis* larvae were placed in a Petri dish with four 1.0 cm^2 leaf disks, two treated with the test substance and two were controls with solvent only; six Petri dishes were used for each extract tested. Each bioassay was terminated after consumption of about 75% of the control disks. Feeding inhibition was calculated as $\%\ FI = [1 - (T/C) \times 100]$, where T is the consumed leaf area of treated and C is the consumed leaf area, from the leaf disks. Also, for this bioassay 10 *Myzus persicae* adults were placed in an agar-coated box with 10 µL of the test substance on the upper surface, obtaining a total of 20 boxes per compound tested. The bioassays were finished after 24 h. The bioassay data was noted based on the number of aphids settled on the treated and control disks. The results from both bioassays were statistically evaluated by the Wilcoxon signed rank test (Burgueño *et al*., 2008).

For these bioassays it was necessary to use the organic phase of the hydrolate obtained in the steam distillation, which was extracted by a solid-phase extraction process (González *et al*., 2013).

Phytotoxic activity: For the phytotoxic activity determination, experiments were conducted with *Lactuca sativa* and *Lolium perenne* seeds on 12-well microplates with 500 µl from pure hydrolate. Germination was monitored daily during 7 days. Radicle length was measured at the end of the bioassay. Twenty-five digitalized radicles were randomly selected for each test with the software Image J Version 1.37r. Results were evaluated using a Mann-Whitney U Test (Julio *et al*., 2016; Martín *et al*., 2011).

Results and Discussion

Essential oil yield: Table 1 shows the average yields of essential oil on a dry weight basis obtained at the end of the extraction processes performed at laboratory scale. The highest yield (0.389%) was achieved with a steam flow of 4.33 $cm^3 \cdot min^{-1}$ and the lowest bed porosity level (0.79). The ANOVA indicates that steam flow, bed porosity, and their interaction are significant factors.

No previous reports regarding the yield of essential oil from J. curcas leaves were found. Therefore, the results obtained in this work were compared against another species of Jatropha, Jatropha gossypifolia, for which another study obtained a 0.87% extraction yield (Aboaba *et al.,* 2015). Malekydozzadeh *et al.,* (2012) used three different steam flow (4000, 7000 and 9000 $cm^3 \cdot min^{-1}$) applied to leaves of Rosmarinus officinialis, where the results indicated that the highest extraction yield of essential oil was obtained under the lowest assessed steam flow (4000 $cm^3 \cdot min^{-1}$) with the lowest superficial velocity and the highest residence time. The same authors also mention that decreases in the steam flow and the compaction of the sample lead to an increase in the extraction yield of essential oil. In the same way, Cerpa 2007, used three steam flows (9, 16 and 24 $cm^3 \cdot min^{-1}$) on leaves and flowers of Rosmarinus officinialis, where the highest yield was obtained with the middle steam flow rate of 16 $cm^3 \cdot min$. Masango *et al*., 2005 also utilized three values of steam flow (2.5, 5 and 20 $cm^3 \cdot min^{-1}$) on leaves of Artemisia annua, where the extraction yield of essential oil was increased by reducing the steam flow.

Residence time is inversely proportional to steam flow, where residence time is the result of dividing the volume of a cylinder of matter by the steam flow; a high steam flow tends to reduce the residence time of the steam flow in the column, leading to a poor extraction of essential oils (Richter *et al.,* 1999; Acedo-Sánchez, 2006). Thus, a balance must exist between steam flow and residence time. However, a high residence time obtained with a very low steam flow rate not provide sufficient energy for extraction of the essential oil. There may be an optimum steam flow rate, with good superficial velocity and appropriate residence time allowing good separation of substances and reducing thermal degradation of the compounds, as flows higher than required will cause steam waste, while flows lower than required will result in low yields of extraction of essential oils (Isbell & Cermak *et al.,* 2004; Cerpa, 2007; Padilla *et al.,* 2007).

Some studies concluded that the highest essential oil transfer took place when the apparent bed density was high (Cerpa, 2007; Malekydozzadeh *et al*., 2012). If the vegetable matter is not correctly compacted, canalization may cause steam to drift sideways, decreasing the contact surface (Arango *et al*., 2012). Also, a low bed density makes steam flow through more open channels with lower resistance, leading to inappropriate contact with the vegetable matter and, therefore, results in a lower extraction yield of essential oil (Özek, 2012).

Chemical profile of essential oil isolated from *J. curcas*: Table 2 shows the preliminary analysis on essential oil composition of *J. curcas* leaves. The essential of extracted at laboratory-scale had 15 compounds, and the majoritarian components were dibutyl phthalate, phytol, and diisooctyl phthalate.

Phytol has been reported to possess antimicrobial activities, toxicity on mouse skin cells and a high repellent activity against *Anopheles gambiae* (Nerio *et al*., 2012; Ghaneian *et al*., 2015).

Research indicates that dibutyl phthalate has antimicrobial and algicidal activities (Bie *et al*., 2012). In recent years there has been much controversy about phthalates in plants, Ruikar *et al*., (2011) and Khatiwora *et al*., (2012) mentioned that phthalates belongs to the secondary metabolites of as *Mimusops elengi* and *Ipomoea carnea* respectively. One of the fraction isolated from *Oreosyce africana* Hook. Leaves was the most important compound was dibutyl phthalate (Bekele *et al*., 2016). More specifically for *J. curcas* presence of phthalates in toxic and non-toxic leaves were detected (Pereira *et al*., 2017; Khan *et al*., 2009). However, it was possible that the plant had accumulated phthalates that were exposed to polluted wastewater (Manayi *et al*., 2014).The resulting table makes it easy to see that the composition of the profile was kept constant. Values above 800 to match and R. match indicate that the mass spectrum corresponds to a good match factor with the library spectrum, which gives reliability to the assignment made (Baharum *et al*., 2010; Sparkman *et al*., 2011) All the samples reported in Table 2, had values of the match factor greater than 800, leading to high confidence intervals based on their probabilities and spectral match scores.

Table 1. Yields (%) and statistics at laboratory scale and pilot scale applied to *Jatropha curcas*.

Experiment	Q ($cm^3 \cdot min^{-1}$)	ε (%)	U ($cm \cdot min^{-1}$)	R_t(min)	Y_{exp}(SD) (%)
EJ1	0.73	0.90	0.05	457.5	0.202 (0.008)^{d}
EJ2	1.78	0.90	0.11	187.6	0.109 (0.002)^{bc}
EJ3	4.33	0.90	0.27	77.1	0.128 (0.004)^{c}
EJ4	8.10	0.90	0.51	41.2	0.031 (0.001)^{a}
EJ5	0.73	0.79	0.05	457.5	0.089 (0.001)^{b}
EJ6	1.78	0.79	0.11	187.6	0.102 (0.001)^{b}
E7	4.33	0.79	0.27	77.1	0.389 (0.009)^{e}
EJ8	8.10	0.79	0.51	41.2	0.010 (0.001)^{a}

Q: Steam flow $cm^3 \cdot min^{-1}$
ε: Bed porosity
U: Superficial velocity $cm \bullet min^{-1}$
R_t: Residence time min
$Yexp$: Experimental yield obtained at the end of process (% mean)
SD: Standard deviation
aMeans followed by the same letter showed no significant difference with Tukey's Test ($p<0.05$)

Table 2. Tentative identification of the essential oils of *Jatropha curcas* obtained at laboratory scale.

Peak	RT	Compound	Match	R. Match	Area%
1	9.7	Thymol	913	929	1.46
2	9.8	Carvacrol	940	940	15.44
3	10.2	Decanoic acid	826	906	1.31
4	11.2	Phenol, 3-(1,1-dimethylethyl)-4-methoxy-	827	856	3.63
5	11.7	Dihydroactinidioliden	843	870	3.55
6	12.7	Blumenol C	841	894	5.19
7	13.3	2-Cyclohexen-1-one, 4-hydroxy-3, 5, 5-trimethyl-4-(3-oxo-1-butenyl)-	803	844	3.87
8	13.4	2-Pentadecanone, 6, 10, 14-trimethyl-	852	928	6.67
9	14.2	Dibutyl phthalate	958	962	22.19
10	15.2	Phytol	891	914	19.2
11	21.0	Diisooctyl phthalate	914	944	17.57

R. T: Retention time (min)

Area %: Area of each compounds are expressed as means. Means were obtained in duplicate

Table 3. Antifeedant activity against *Spodoptera littoralis* and *Myzus persicae.*

Test substance	Population	
	***S. littoralis*[a]**	***M. persicae*[c]**
EJ7	71.31 (27.662)[b]*	79.17 (26.494)[d]*

[a]Percent settling inhibition

[b]The values are the mean (SD) of six replicates

[c]Percent feeding inhibition ($\%FI/\%SI = [(1 - (T/C)) \times 100]$ where T, consumption in settling on treated disk), and C, consumption in settling on control disk

[d]The values are the mean ± SD of twenty two replicates

* p<0.05, Wilcoxon paired rank test

Antifeedant bioassay: Due to the low essential oil quantities the organic phase of the EJ7 hydrolate (the experiment with the highest yield) was used in all the bioassays.

Table 3 shows the bioactivity of the hydrolate, where a value greater than 75% is considered an active extract (Burillo & González, 2009). In this case, the extract was active only against *M. persicae*.

In another study, the methanolic leaf extract of *J. curcas* with 5% concentration achieved the highest mortality at 60% against third instar larvae of *Spodoptera litura*, which belonged to the genus *Spodoptera* (Ingle *et al*., 2017), while in this study an inhibition of sixth instar larvae of *Spodoptera littoralis* was observed with 71.31%, from essential oil of *J. curcas*.

It had been reported that *J. curcas* seed oil and petroleum ether extracts from *J. curcas* leaves were effective against *M. persicae* (Holtz *et al*., 2016: Devi *et al*., 2008). In this work *J. curcas* essential oil was effective against *M. persicae* with 79.17%, while Devi *et al*., (2008), mentioned a bioactivity with 60%.

Phytotoxic activity: Table 4 shows the results of the phytotoxic effects of the hydrolate of *J. curcas*. It was observed that the hydrolate had allelopathic effects on seed germination, nevertheless *L. perenne* was more sensitive than *L. sativa*. The extracts had the greatest allelopathic effect on the roots, as the allelopathic compounds induced the development of abnormal seedlings (Reichel *et al*., 2013; Syed & Shinwari, 2016).

Another study showed that aqueous extracts of *J. curcas* leaves and roots inhibited the growth of *Zea mays* and *Nicotiana tabacum*. It has been shown that ethanolic and methanolic extract of dried jatropha leaves affected the seedling growth of wheat (*Triticum aestivum*), being the ethanolic extract the most phytotoxic (Reichel *et al*., 2013). Antonelli *et al*., (2015), show that aqueous extract of *J. curcas* leaf was allelopatic effect against *Brassica oleracea*. Allelopathic effects of Jatropha leachates leaves on wheat seedlings has also been demonstrated (Tomar *et al*., 2015). However, aqueous extracts at lower concentrations promoted the germination and growth of wheat seeds, due to the phenolic compounds present in the extract, which possibly act as a bioregulator (Khattak *et al*., 2015).

Table 4. Phytotoxic effects against Lactuca sativa and Lolium perenne.

Test substance	Population				
	L. sativa		L. perenne		
	Germination[a]	Growth[b]	Germination[a]	Growth[b]	Growth[b]
	168 h	root	168 h	rooth	leaf
EJ7	95.00 (5.77)[b]	34.84 (0.24)[b]*	64.10 (28.72)[b]*	30.60 (1.07)[b]*	59.88 (1.64)[b]*
Control[c]	100.00 (0.00)[b]	100.00 (0.00)[b]	100.00 (5.00)[b]	100.00 (0.90)[b]	100.00 (0.51)

[a]Percent control

[b]The values are the mean (SD) of four replicates

[c]Positive control

*p< 0.05, Mann−Whitney U-test

Conclusions

No previous studies regarding the extraction of essential oil from *J. curcas* leaves were found by steam distillation, this implies that this is the first report on the chemical composition of this essential oil.

To obtain higher yields, an adequate steam flow must be obtained, maintaining a sufficient *residence* time to extract all the available oil from the glandular trichomes, in addition to compacting the sample for achieving a greater transfer of oil to the steam.

References

Aboaba, S.A., M.A. Adebayo and O. Olayiwola. 2015. Volatile constituents of *Jatropha gossypifolia* L. grown in Nigeria. *AJEONP*., 2(4): 8-11.

Acedo Sánchez, J. 2006. . Medidas de caudal. In: Instrumentación y control avanzado de procesos. Ed. Díaz de Santos. España, pp. 15

Ahirrao, R.A., M.R. Patel, D.M. Pokal, J.K. Patil and H.P. Suryawanshi. 2011. Phytochemical screening of leaves of *Jatropha curcas* plant. *IJRAP*., 2(4): 1324-1327.

Antonelli, J., C. Lindino, R. Baricciatti, S. Melegari, W. Nadaletti, P. Cremonez and E. Rossi. 2015. Allelopatic effect of irrigation with different concentrations of leaf extracts of *Jatropha curcas* L. on growth Brassica oleracea. *Afr. J. Agric. Res*., 11(9): 779-782.

Arango, B.O., F. Bolaños, O. Villota, A. Hurtado and I. Toro. 2012. Optimización del rendimiento y contenido de timol de aceite esencial de orégano silvestre obtenido por arrastre con vapor. *Revista Biotecnología en el sector Agropecuario y Agroindustrial*, 10(2): 217-226.

Baharum, S.N., H. Bunawan, M.A. Ghani, W.A. Mustapha and N.M. Noor. 2010. Analysis of the chemical composition of the essential oil of *Polygonum minus* Huds. using two-dimensional gas chromatography-time-of-flight mass spectrometry (GC-TOF MS). *Molecules*, 15(10): 7006-7015.

Bekele, D., H. Tekie, Z. Asfaw and B. Petros. 2016. Bioactive chemical constituents from the leaf of *Oreosyce africana* Hook. f (Cucurbitaceae) with mosquitocidal activities against adult *Anopheles arabiensis*, the Principal Malaria Vector in Ethiopia. *J. Fertil. Pestic*., 7(159): 1-8.

Bie, C.C., F.M. Li, Y.F. Wang, H.Y. Wang, Y.H. Zhao, W. Zhao and Z.Y. Wang. 2012. Mechanism of the Inhibitory Action of Allelochemical Dibutyl Phthalate on *Algae Gymnodinium* breve. *Environ. Sci.*, 33: 228-232.

Burgueño, T. E., L. Castillo, A. González and P.J. Nathan. 2008. Antifeedant and phytotoxic activity of the sesquiterpene p-benzoquinone perezone and some of its derivatives. *J. Chem. Ecol*., 34(6): 766-771.

Burillo, J. and C.A. González. 2009. Ensayos con insectos: diseño experimental In: (Eds.): CYTED. Gobierno de Aragón, Centro de Investigación y Tecnología Agroalimentaria Insecticidas y repelentes de insectos de origen natural, España, pp. 16-41.

Calvo, I.L. 2012. *Lippia graveolens*. In Plantas Aromáticas de Yucatán. Mérida, Yucatán, México: Centro de Investigación Científica de Yucatán, pp. 60-65.

Cerpa, C.M. 2007. Hidrodestilacion de aceites esenciales: modelado y caracterización. Doctoral thesis. Universidad de Valladolid (España).

Cerpa, M., R. Mato and M. Cocero. 2008. Modeling steam distillation of essential oils: Application to lavandin super oil. *AIChE*., 54(4): 909-917.

Chauhan, N., P. Kumar, S. Mishra, S. Verma, A. Malik and S. Sharma. 2015. Insecticidal activity of *Jatropha curcas* extracts against housefly. *Musca domestica*. *Environ Sci Pollut Res*., 22(19): 14793-14800.

Devi, P.S., L.R. Devi and I.M. Singh. 2008. Evaluation of plant products against aphid (*Myzus persicae* Sultz) transmission of mosaic disease of leaf mustard (*Brassica juncea* var. rugosa). *Ind. Phytopath*., 61(4): 514-517.

Ghaneian, M.T., M.H. Ehrampoush, A. Jebali, S. Hekmatimoghaddam and M. Mahmoudi. 2015. Antimicrobial activity, toxicity and stability of phytol as a novel surface disinfectant. *EHEMJ*., 2(1): 13-16.

González, C.A., Y.M. Andrés, H.C. Díaz, A.J. Burillo, P.R. Cabrera and U.J. Navarro. 2013. Use of essential oils, supercritical extracts and aqueous residues generated during a process for obtaining organic extracts from the *Artemisia absinthium* L. plant. Patent. España. 19 de Septiembre de 2013, patent number WO 2013/135915 A1.

Holtz, A.M., J.J.P. Marchriio, M.L. Franzin, H.H. Paulo, J.M.C. Botti and T. Coffler. 2016. Potencial de manejo de *Myzus persicae* com óleo de pinhão manso armazenado em diferentes embalagens. *Revista Agrogeoambiental.,* 8(3): 41-50.

Hossain, M.A., R.A. Al-Hashmi, A.M. Weli, Q. Al-Riyami and J.N. Al-Sabahib. 2012. Constituents of the essential oil from different brands of *Syzigium caryophyllatum* L. by gas chromatography-mass spectrometry. *Asian Pac. J. Trop. Biomed*., 2(3): 1446-1449.

Ingle K.P., A.G. Deshmukh, D.A. Padole, M.S. Dudhare, M.P. Mangesh and V.C. Khelurkar. 2017. Bioefficacy of crude extracts from *Jatropha curcas* against *Spodoptera litura*. *J. Entomol. Zool. Stud.*, 5(1): 36-38.

Isbell, T. and S. Cermak. 2004. Purification of meadowfoam monoestolide from polyestolide. *Ind. Crops Prod.,* 19: 113-118.

Juliet, S., R. Ravindran, S.A. Ramankutty, A.K. Gopalan, S.N. Nair, A.K. Kavillimakkil, S. Gosh, A. Bandyopadhyay and S. Gosh. 2012. *Jatropha curcas* (Lin) leaf extract – a posible alternative for population control of *Rhipicephalus* (Boophilus) annulatus. *Asian Pac. J. Trop. Dis*., 2(3): 225-229.

Julio, L.F., A.F. Barrero, M.M. Herrador del Pino, J.F. Arteaga, J. Burillo, M.F. Andres and C.A. González. 2016. A. Phytotoxic and nematicidal components of *Lavandula luisieri*. *J. Nat. Prod*., 79(2): 261-266.

Khan, A.L., J. Hussain, M. Hamayun, Z.K. Shinwari, H. Khan, Y.H. Kang, S.M. Kang and L.J. Lee. 2009. Inorganic profile and allelopathic effect of endemic *Inula koelzii* from Himalaya Pakistan. *Pak. J. Bot.*, 41(5): 2517-2527.

Khatiwora, E., V.B. Adsul, M. Kulkarni, N.R. Deshpande and R.V. Kashalkar. 2012. Anti-bacterial activity of dibutyl phthalate: a secondary metabolite isolated from *Ipomoea carnea* stem. *J. Pharm. Res*., 5: 150-2.

Khattak, A., F. Ullah, S.M. Wazir and Z.K. Shinwari. 2015. Allelopathic potential of *Jatropha curcas* L. leaf aqueous extracts on seedling growth of wheat. *Pak. J. Bot*., 47(6): 2449-2454.

Kotnik, P., M. Škerget and Z. Knez. 2007. Supercritical fluid extraction of chamomile flower heads: Comparison with conventional extraction, kinetics and scale-up. *J. Supercrit. Fluid*, 43(2): 192-198.

Koul V. K., B.M. Gandotra, S. Koul, S. Ghosh, C.L. Tikoo and A.K. Grupta. 2004. Steam distillation of lemon grass (*Cymbopogon* spp.). *Indian J. Chem. Technol*., 11: 135-139.

Kovendan, K., K. Murugan, S. Vincent and S. Kamalakannan. 2011. Larvicidal efficacy of Jatropha curcas and bacterial insecticide, *Bacillus thuringiensis*, against lymphatic filarial vector, *Culex quinquefasciatus* Say (Diptera: Culicidae). *Parasitol Res*., 109(5): 1251-1257.

Ma, H.F., N.J. Lang, L.P. He, Z. Yu, K. Zheng, M. Peng, Z. Xiang, J.J. Kong. R.L. and Yuan. 2011a. Volatile components from plant parts of *Jatropha curcas* in Jianshui, Yunnan Province. *Journal of Zhejiang A and F University,* 28: 674-679.

Ma, Y., J. Chun, S. Wang and F. Chen. 2011b. Allelopathic potential of *Jatropha curcas*. *Afr. J. Biotechnol*., 10(56): 11932-11942.

Malekydozzadeh, M., P.P. Khadiv, Sh. Rezazadeh, H. Abolghasemi, Z. Salehi and Q. Li. 2012. Application of multistage steam distillation column for extraction of essential oil of *Rosemarinus officinialis* L. *IAChE.*, 9(4): 54-64.

Manayi, A., M. Kurepaz-Mahmoodabadi, A.R. Gohari, Y. Ajani and S. Saeidnia. 2014. Presence of phthalate derivatives in the essential oils of a medicinal plant *Achillea tenuifolia*. *Daru U J. Pharm. Sci*., 22(1): 78.

Martín N.L., L. Julio, J. Burillo, J. Sanz, A. Mainar and C.A. González. 2011. Comparative chemistry and insect antifeedant action of traditional (Clevenger and Soxhlet) and supercritical extracts (CO2) of two cultivated wormwood (*Artemisia absinthium* L.) populations. *Ind. Crops Prod*., 34 (2011): 1615-1621.

Martín, N.L. 2012. Aplicación de procesos de separación con CO2 supercrítico a la producción y optimización de bioplaguicidas. Doctoral thesis. Universidad de Zaragoza. Zaragoza (España).

Masango, P. 2005. Cleaner production of essential oils by steam distillation. *Jclepro.*, 13(8): 833-839.

Nerio, L.S., J. Olivero, E.E and Stashenko. 2012. Repellent activity of essential oils: A review. Bioresour. *Technol*., 101(1): 372-378.

Ordaz, G., H. D'Armas, D. Yáñez and S. Moreno. 2011. Composición química de los aceites esenciales de las hojas de *Helicteres guazumifolia* (Sterculiaceae). *Piper tuberculatum* (Piperaceae). *Scoparia dulcis* (Arecaceae) y *Solanum subinerme* (Solanaceae). recolectadas en Sucre. Venezuela. *Rev. Biol. Trop*., 59(2): 585-595.

Özek, T. 2012. Distillation parameters for pilot plant production of *Laurus nobilis* essential oil. *Rec. Nat. Pro.*, 6(2): 135-143.

Pabón, L.C. and P. Hernández. 2012. Importancia química de *Jatropha curcas* y sus aplicaciones biológicas, farmacológicas e industriales. *Rev. Cubana Plant. Med.*, 17(2): 194-209.

Padilla, M. F., O. González, R. Prado, H. Gutiérrez, E.M. Estarrón, G.H. Vega and R.J. Padilla. 2007. Nuevo equipo y proceso de destilación fraccionada en continuo por arrastre con vapor de aceites esenciales del jugo de limón mexicano. *e-Gnosis Revista Digital Científica y Tecnológica,* 5: 1-16.

Pereira, I., T. Carvalho, R. Wanderson, P.R. Filgueiras, B.G. Laviola, C.M. Rodrigues, P.V. Abdelnur and B.G. Vaz. 2017. Differentiation of toxic and non-toxic leaves of *Jatropha curcas* L. genotypes by leaf spray mass spectrometry. *J. Braz. Chem.*, 28(8): 1461-1466.

Rabha, B., R. Gopalakrishnan, I. Baruah and L. Singh. 2012. Larvicidal activity of some essential oil hydrolates against dengue and filariasis vectors. *J. Med. Res*., 1(1): 14-16.

Rajeswari, G., M. Murugan and V.R. Mohan. 2012. GC-MS analysis of bioactive components of *Hugonia mystax* L. (Linaceae). *J Pharm. Biomed.*, 29(29): 818-824.

Reichel, T., J. Barazetti, S. Stefanello and P.D.C. Zonetti. 2013. Allelopathy of leaf extracts of Jatropha (*Jatropha curcas* L.) in the initial development of wheat (*Triticum aestivum* L.). *Revista IDESIA (Arica) Chile*, 30: 45-52.

Richter, P., M. Toral and R. Manriquez. 1999. Sistema de extraccion liquido-liquido en continuo con separador de fases tipo sandwich para la determinacion de cobre al estado del complejo cobre(i)-batocuproina. Boletín de la Sociedad Chilena de Química., 44: 451-458.

Ruikar A.D., T.V. Gadkari, U.D. Phalgune, V.G. Puranik and N.R. Deshpande. 2011. Dibutyl phthalate, a secondary metabolite from *Mimusops elengi*. *Chem. Nat. Compd*., 46(6): 955-956.

Salomón, I., H.O. López, C. García, S.M. González and V. Moreno. 2009. Desarrollo de una tecnología para la obtención de extracto acuoso de hojas de *Morinda citrifloia* L. (noni*). Rev Cubana Plant Med.*, 14(2): 1-8.

Sparkman, D., Z. Penton and G.K. Fulton. 2011. Masa spectral data interpretation. In gas chromatography and mass spectrometry (Second Edition). A Practical Guide. Academic Press. The Boulevard, Langford Lane, Kidlington, Oxford OX51GB, UK, pp. 149-178.

Stashenko, E.E. 2009. Aceites esenciales. Edit. División de Publicaciones UIS. Bucaramanga – Santander, Colombia, pp. 13-14.

Syed, K. and Z.K. Shinwari. 2016. Allelopathic effect of methanolic extracts of genetically modified and non-genetically modified canola on soybean. *Toxicol. & Indus. Health*, 32(3): 564-575.

Tomar, N.S., M. Sharma and R.M. Agarwal 2015. Phytochemical analysis of *Jatropha curcas* L. during different seasons and developmental stages and seedling growth of wheat (*Triticum aestivum* L) as affected by extracts/leachates of *Jatropha curcas* L. *Physiol. Mol. Biol. Plants*, 21(1): 83-92.

Tomass, Z., M. Hadis, A. Taye, Y. Mekonnen and B. Petros. 2011. Larvicidal effects of *Jatropha curcas* L. against *Anopheles arabiensis* (Diptera: Culicidea). *MEJS.*, 3(1): 52-64.

(Received for publication 18 January 2018)