

New Chemical Compounds Isolated from the Stem Bark of *Talipariti elatum* Sw. in Cuba

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Abstract: Stem Bark of *Talipariti elatum* Sw. was extracted by Soxhlet with toluene. After extracts concentration, the sample was processed with CHCl_3 that allow getting a precipitate. The phytochemical studies of stem bark of *Talipariti elatum* have led to the identification of two triterpenoids: friedelin and fridelinol, an esterified triterpenoid, three alcohols and one aromatic acid. The structures of these compounds were identified by analysis of GC/MS data and comparison with values of literature.

Keywords: *Talipariti elatum*, Toluene, Soxhlet, Triterpenoids, GC/MS.

1. INTRODUCTION

Talipariti elatum S.w Fryxell (Malvaceae), a tree with a wide distribution in Cuba, that growth in any type of soil particularly in swanpyones. It can get about 25 m of height. It shows peciolated leaves, to mentose-velvet like and heart-shaped at the basis. The flowers are big, yellow flower leaves that become reddish when old. The flowers are used as appetitive, emollient, sudorific and as excellent pectoral combined with the flowers of *Hibiscus rosa-sinensis*. The mixture is used in traditional medicine as expectorant and antasthmatic (Roig, 2014). It is reported that flowers of *T. elatum* contains flavonoids (quercetin and rutin), sugars, phenols and tannins, triterpens and steroids and saponins (Acosta & Rodríguez, 2006), although the plants' composition has not been determined in detail so far. Gossypetin -3'-O-glucoside was isolated for the first time from the flowers of the plant in Martinic Island by maceration with methanol (24 h), and Soxhlet extraction with methanol, ethyl acetate and 1,2-dimethoxyethane as solvents (François-Haugrin et al., 2015) and in Cuba in 2016 (Yaque et al., 2016).

Gas chromatography coupled with mass spectrometry (GC-MS) in particular is a fast and accurate method widely applied in diagnostics, functional genomics and for screening purposes. Following solvent extraction and derivatization, hundreds of metabolites from different chemical groups can be characterized in one analytical run. Gas chromatography (GC) in particular is characterized by sensitivity and reliability of separations and detection of complex sample mixtures (Wang et al. 2015). Coupling with mass spectrometry (MS) provides highly robust analysis platforms compared to liquid chromatography (LC-MS) and allows for the identification of compounds based on the use of commercially or publicly available MS libraries and resources in combination with retention time index (RI) data (Rohloff, 2015). The aim of this research was to characterize the chemical components present in the toluene extracts of stem bark from *T. elatum* S.w.

2. MATERIAL AND METHODS

2.1. Plant Material

Stem bark was collected in January 2017 in the gardens of the Faculty of Pharmacy and Foods at Havana University, and identified at the herbarium of National Botany Garden of Havana, where the voucher specimen no. HAJB 82587 has been deposited. Specimen is registered as *Talipariti elatum* Sw. (Fryxell) Malvaceae (Sin. *Hibiscus elatus* Sw).

2.2. Extract and Samples Preparation

The sample was cleaned twice with distilled water. Then the sample was dry in an oven with controlled temperature at 40 °C during 5 days. After dried, the sample was pulverized to powder using a mechanical grinder. The particles were uniformed using a sieve (1 mm) (Fig. 1). The powder was stored in an air-dried packet. The extracts were prepared with the ground material (60 g) without screen extracted in a Soxhlet apparatus with 675 mL of toluene during 16 hours. The extracts were concentrated and evaporated under vacuum to 200 mL at 120 rpm, a temperature of 70°C and 500 mbar.



Fig.1. Powdered drug

After concentration the extract was dissolved in 20 mL of chloroform and added slowly over 100 mL of a mixture of ketone-methanol (1:1). Trying to rich a second dissolution in chloroform a cream yellow precipitate was collected by filtration on a filter of porosity 3, and dried in the dark at room temperature. After 24 hours another cream yellow precipitate appear and was recovered and purified under same conditions (Fig. 2). That product was used in characterization process.

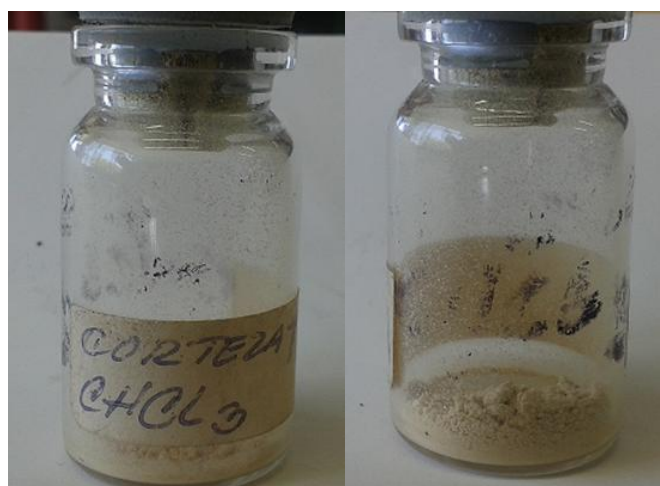


Fig. 2. Precipitate insoluble in $CHCl_3$.

2.3. Procedures, Instrumentation and Parameters

The sample were subjected to chromatographic analysis in equipment GC/MS, brand Shimadzu QP2010, equipped with a splitter split/splitless. With a BP5 (30 m × 0.25 mm × 0.25 microns) capillary column under the following chromatographic conditions: Helium gas carrier obtained by electron impact fragments to a power of 70 eV rate of 1.2 mL/min, 1:50 split flow and the volume of injected sample of 1 ul. Programmed oven temperature: initial temperature was 70°C with a heating ramp of 10°C/min to 300°C and remained stable at this temperature for 10 minutes. Subsequently the temperature was increased at a rate of 10°C/minute to 300°C for a total time of 78 minutes with an injector temperature 250°C and the interface temperature 300°C. The compounds were analyzed using GC/MS NIST21 and NIST107 library and having into account the results obtained after phytochemical screening according with Gómez (2017). Silylation agent was N, O-bis (trimethylsilyl) trifluoroacetamide (BSTFA) CAS 25561-30-2 Lot: 0901-1 Macherey-Nagel GmbH & C. KG. Chromatographic running was done twice, with and without BSTFA.

3. RESULTS AND DISCUSSION

Chromatographic profile of both running showed the same amount of chemical components with few differences that corresponding to adduct formed in the derivatization process (Figures 3 and 4). For that reason the authors determined to analyze the sample without BSTFA and because of the most of compounds were found between 35 and 65 minutes in both cases (Fig. 5).

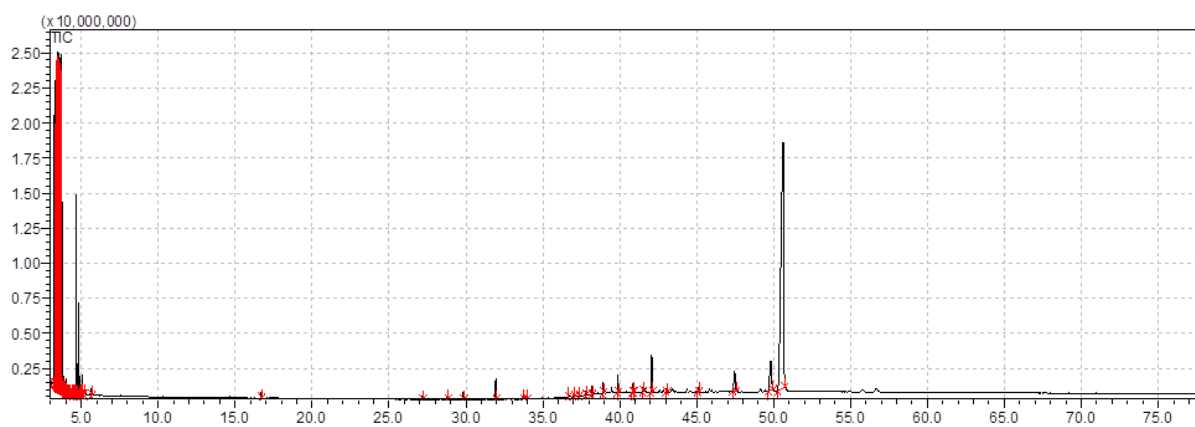


Fig. 3. Chromatographic profile of the sample with BSTFA.

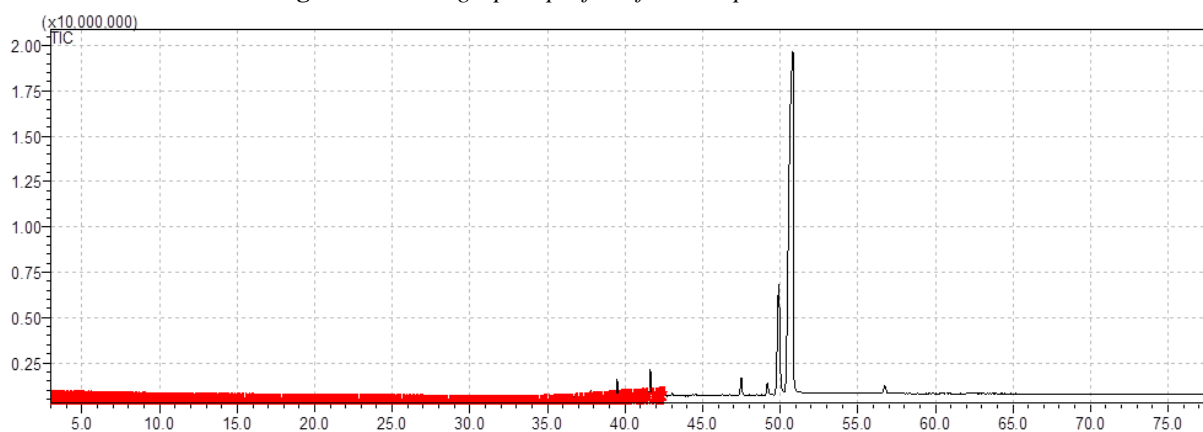


Fig.4. Chromatographic profile of the sample without BSTFA.

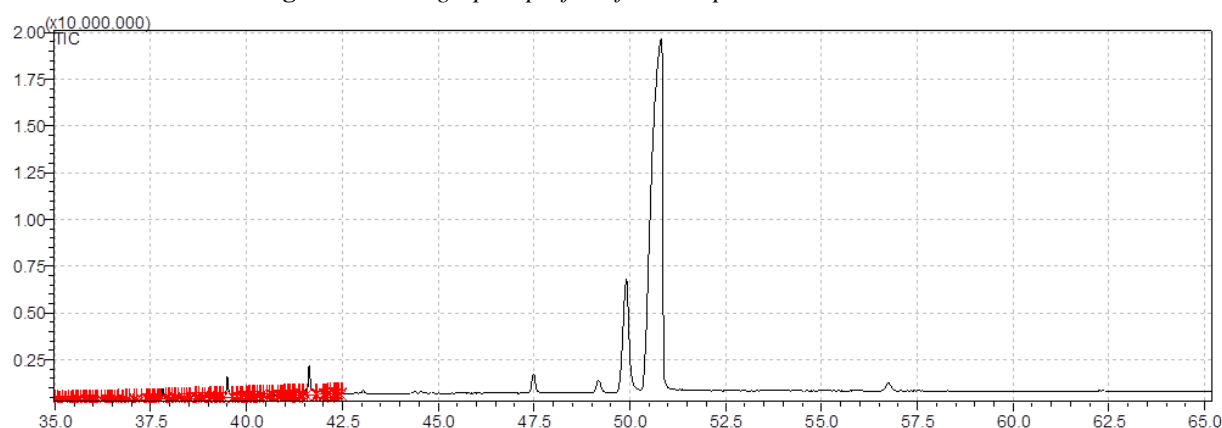


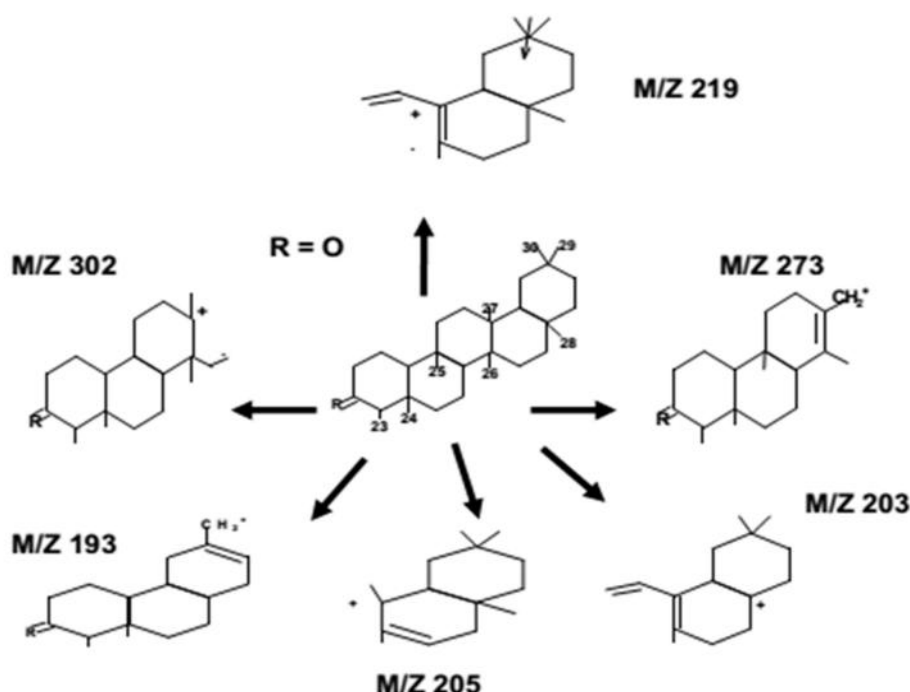
Fig. 5. Current chromatogram without BSTFA (Rt-35-65 min).

Only eight compounds were identified by GC/MS from the insoluble sample in CHCl_3 . The three first chemical metabolites are related with aliphatic alcohols with 27, 18 and 20 carbon atoms respectively: 1-Heptacosanol, 1,2-Octadecanediol and 1-Eicosanol. Another compound related with alcohols was identified as 1-Naphthalenopropanol, α -ethyldecahydro- α -,5,5,8a-tetramethyl-2methylene-, [1S-[1. α . Three secondary metabolites stretchy related were identified as Acetic acid, 4,4a, 6b, 8a, 11, 11, 12b, 14a-octamethyl-3-oxodocosahydropicen-2-yl ester and two members of the Friedelane series: Friedooleanan-3-ol and Friedelin (Friedelan-3-one). The last component was identified as 4-tert-Butylphenoxy- α -Propionic acid. Their corresponding acquired data are shown in Table 1.

Table 1. GC/MS spectral data found in the solid sample of stem bark from *T. elatum*.

No.	Rt (min)	Compound name	Mol. Form.	Mol. Weight	Base Peak
1	37.814	1-Heptacosanol	C ₂₇ H ₅₆ O	396	43
2	39.512	1,2-Octadecanediol	C ₁₈ H ₃₈ O ₂	286	57
3	41.643	1-Eicosanol	C ₂₀ H ₄₂ O	298	57
4	47.510	1-Naphthalenepropanol	C ₂₀ H ₃₆ O	292	137
5	49.186	Acetic acid, 4,4a, 6b....	C ₃₂ H ₅₂ O ₃	484	43
6	49.931	Friedooleanan-3-ol	C ₃₀ H ₅₂ O	428	69
7	50.769	Friedelin	C ₃₀ H ₅₀ O	426	69
8	56.755	4-tert-Butylphenoxy.....	C ₁₃ H ₁₈ O ₃	222	207

Friedooleanan-3-ol and Friedelin (Friedelan-3-one) showed the most prominent peaks indicating that they are the most abundant chemical components in the sample. According to Gomez 2017, there are evidences of the presence of triterpenes and steroids in the extracts of this part of the plant because the test with Liebermann-Burchard was positive (Zhu et al., 2016). It is the first time that both components are reported from the stem bark of *Talipariti elatum* in Cuba. The fragmentations patterns propose for both compounds are similar in general, due to the great coincidence in fragment ion peaks, although the molecular masses are different by two units.

**Fig. 6.** Proposal fragmentation patterns for Friedelan-3 β -ol and Friedelin.

4. CONCLUSIONS

GC/MS was used for the generation of chemical fingerprints and the identification of several compounds in *T. elatum*. The spectral analysis of the solid precipitate recovered from the toluene extract showed the majority of eight compounds. Among them, two pentacyclic triterpenoids were characterized in this part of the plant at the same time: Friedelan-3 β -ol and Friedelin-3-one for the first time. The compounds identified can be also used as biomarkers especially for *T. elatum* because of the little research has been published for this species. Chromatographic profile of the stem bark reveals high predominance of alcohols and triterpenoids. Therefore, stem bark of *T. elatum* may be considered a source of important phytochemicals with bioactive properties to be explored for pharmaceutical applications.

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Citation: J. González et al., " New Chemical Compounds Isolated from the Stem Bark of *Talipariti Elatum* Sw. in Cuba ", *International Journal of Advanced Research in Botany*, vol. 4, no. 1, p. 14-18, 2018. <http://dx.doi.org/10.20431/2455-4316.0401003>

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