

# Digging of Phytochemicals using Mass Spectrometric Machines

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**Associate Professor**

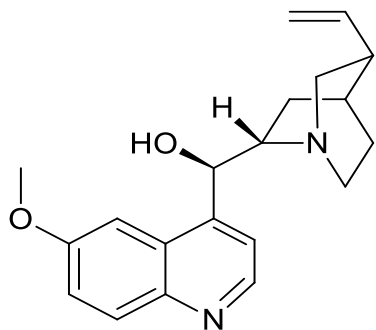
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# Historically Important Natural Products from Plants

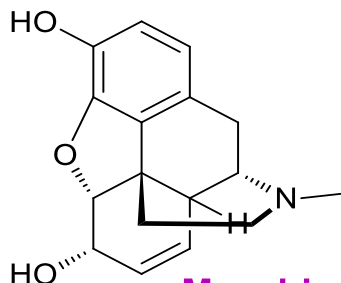


**Quinine**  
**Anti-malarial**

*Cinchona succirubra* Pav.

**Isolation: 1820, Pelletier et al.**

**Synthesis: 1944, Woodward**

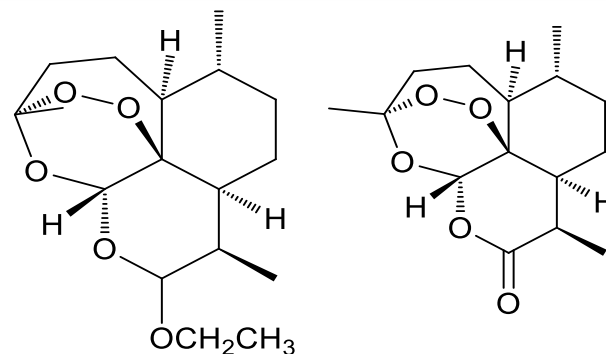


**Morphine**  
**Painkiller**

*Papaver somniferum* L.

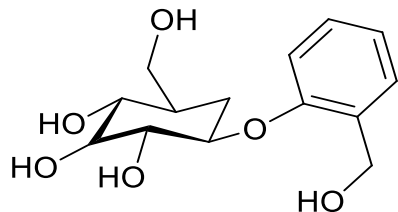
**Isolation: 1806, Sertürner**

**Synthesis: 1954, Ginsberg**



**Arteether** **Artemisinin**  
**Antimalarial drugs**

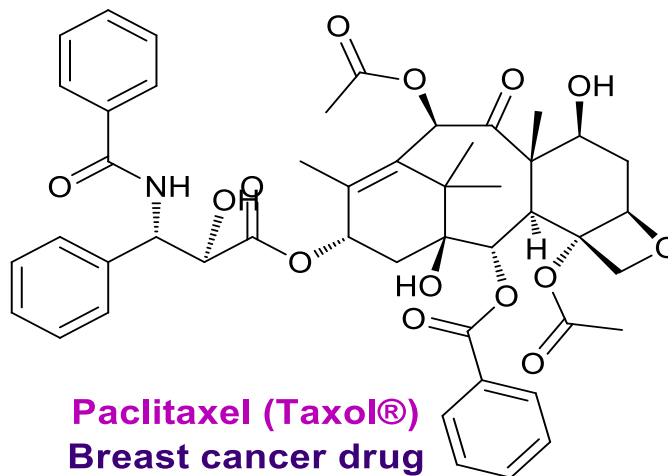
*Artemisia annua*



**Salicin**

*Salix alba* L.

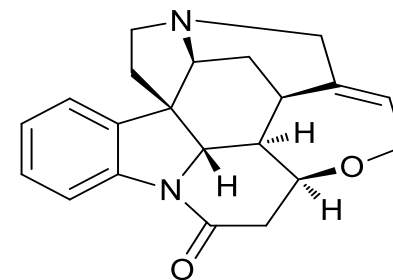
**Structure and synthesis: 1906, Irvine**



**Paclitaxel (Taxol®)**  
**Breast cancer drug**

*Taxus brevifolia*

**Isolation: 1971, Wani et al.**



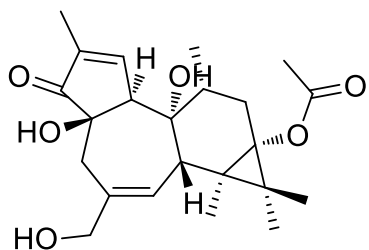
**Strychnine**  
**Pesticide**

*Strychnos nux-vomica*

**Isolation: 1818, Pelletier et al.**

**Synthesis: 1954, Woodward**

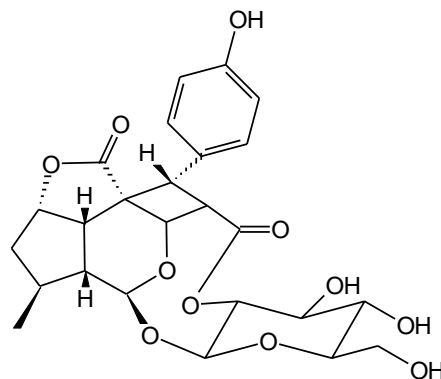
# Historically Important Natural Products from Plants



**Prostratin**

Used for the treatment of (AIDS)

*Homalanthus nutans*



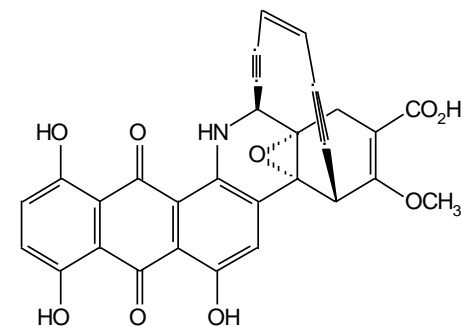
**(-)-Littoralisone**

Neurotrophic Growth Factor

*Verbena littoralis* L

Isolation and structure: 2001, Li

Synthesis: 2005, Mangion



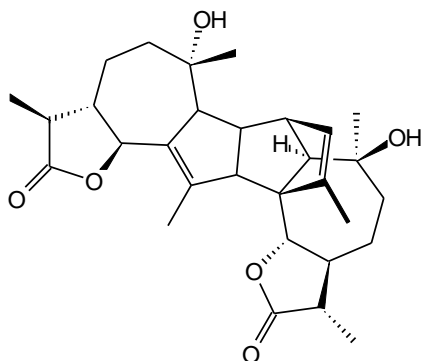
**Dynemicin A**

Antibiotic

*Micromonospora chersina*

Structure: 1989, Matsumoto et al

Synthesis: 1991, Nicolau



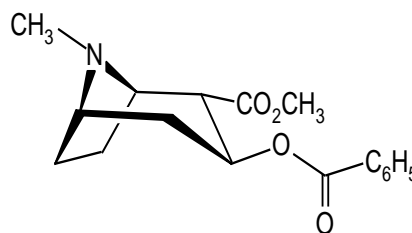
**(+)-Absinthin**

Anti-inflammatory Agent

*Artemisia absinthium* L

Isolation: 1953, Herout

Synthesis: 2004, Zhang



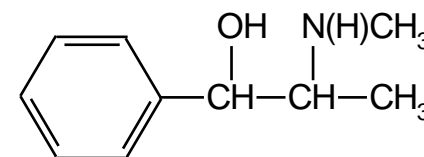
**Cocaine**

Appetite Suppressant

*Erythroxylon coca*

Isolation: 1859, Niemann

Synthesis: 1923: Willstätter



**Ephedrin**

Decongestant

*Ephedra equisetina*

Structure and synthesis:

1920, Späth and Göring

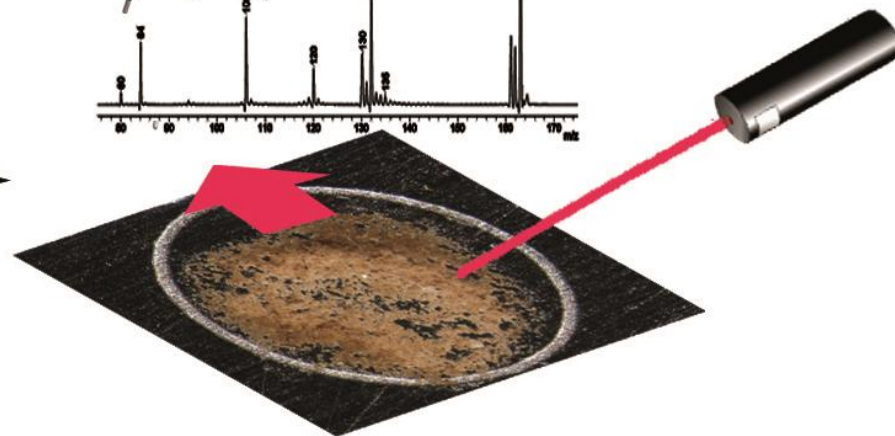
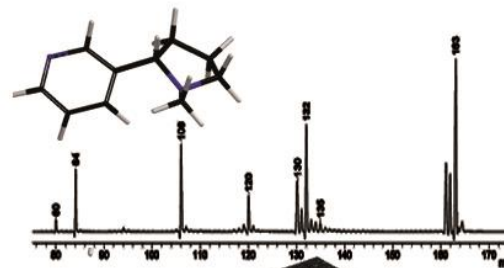
# LDI Analysis of Plant Powderd Materials



1.



2.



Musharraf et al, Journal of American Chemical Society of Mass Spectrometry, (25 (4), 530-537, 2014)

# Solvent Effect: LDI-MS Analysis

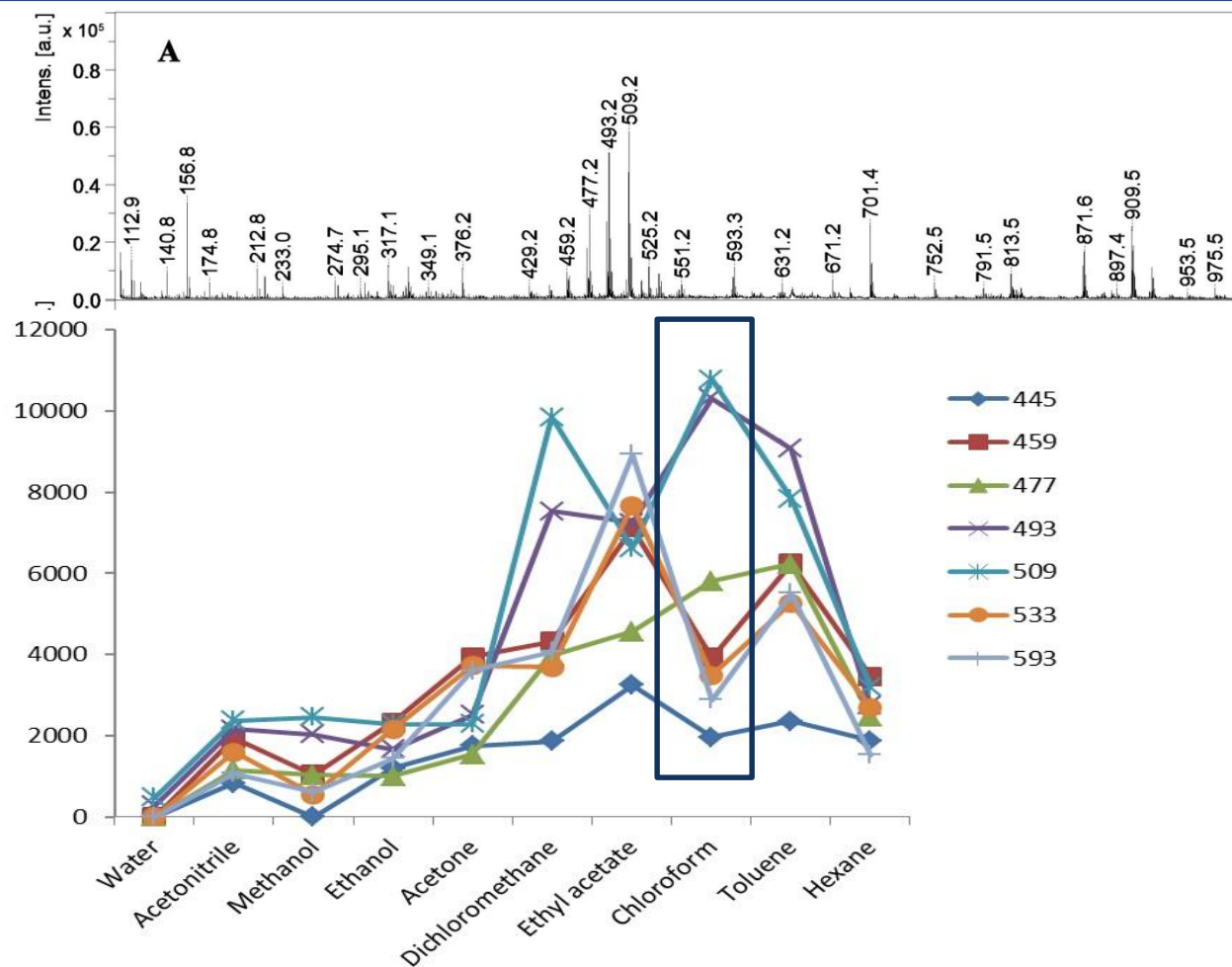
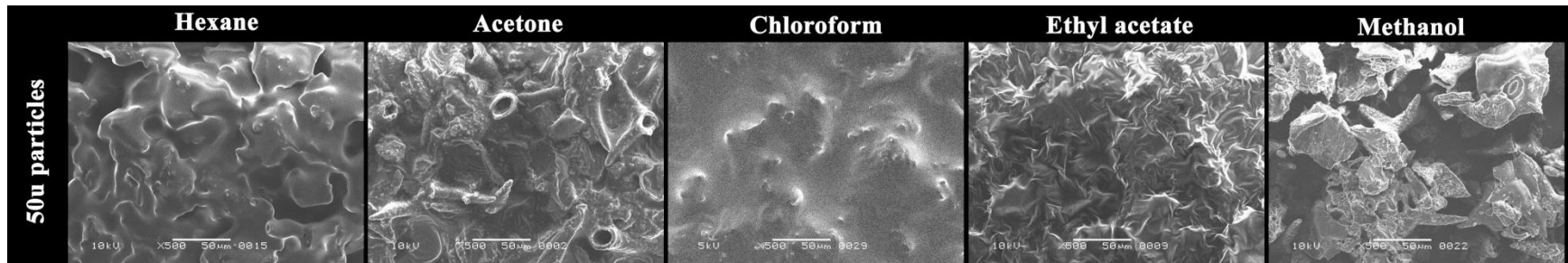
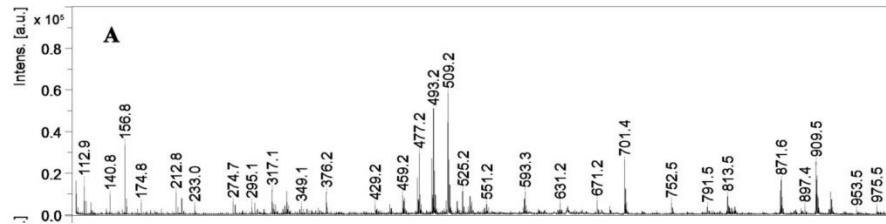


Figure 1. Graph between solvents of various polarities (decreasing order) and the intensity of TOF-MS ions of selected withanolides found in *Withania somnifera* leaf.

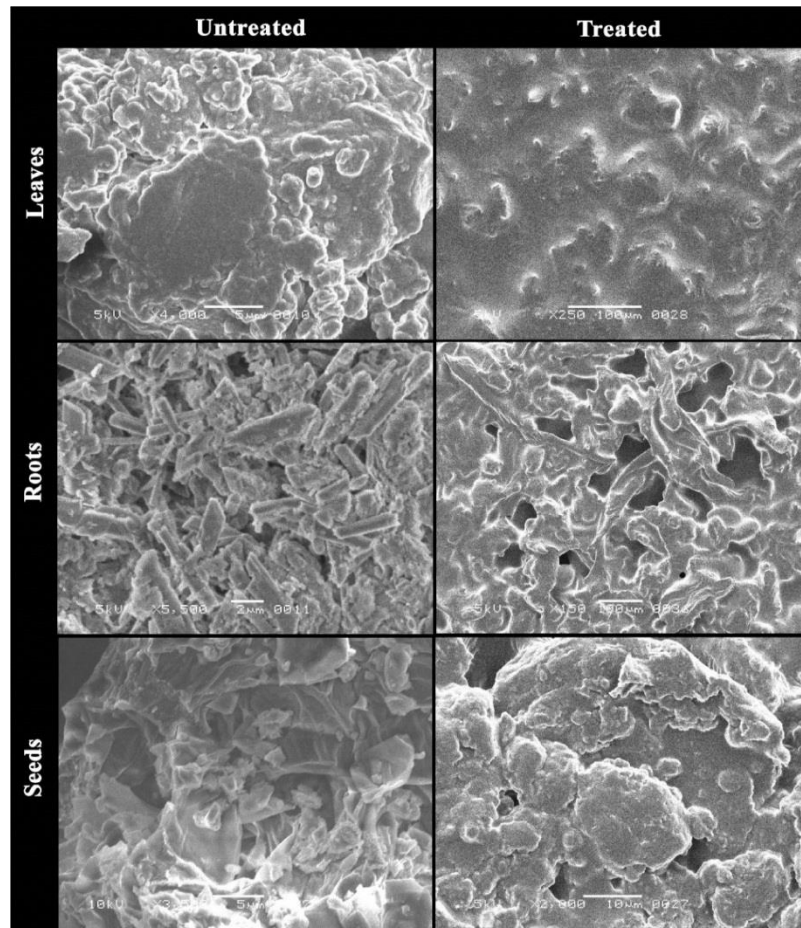
# Solvent Effect: SEM Analysis



**Figure 2.** SEM Images of *Withania somnifera* plant material passed through 50  $\mu$ m mesh sieves treated with different solvents

Musharraf et al, Journal of American Chemical Society of Mass Spectrometry, (25 (4), 530-537, 2014)

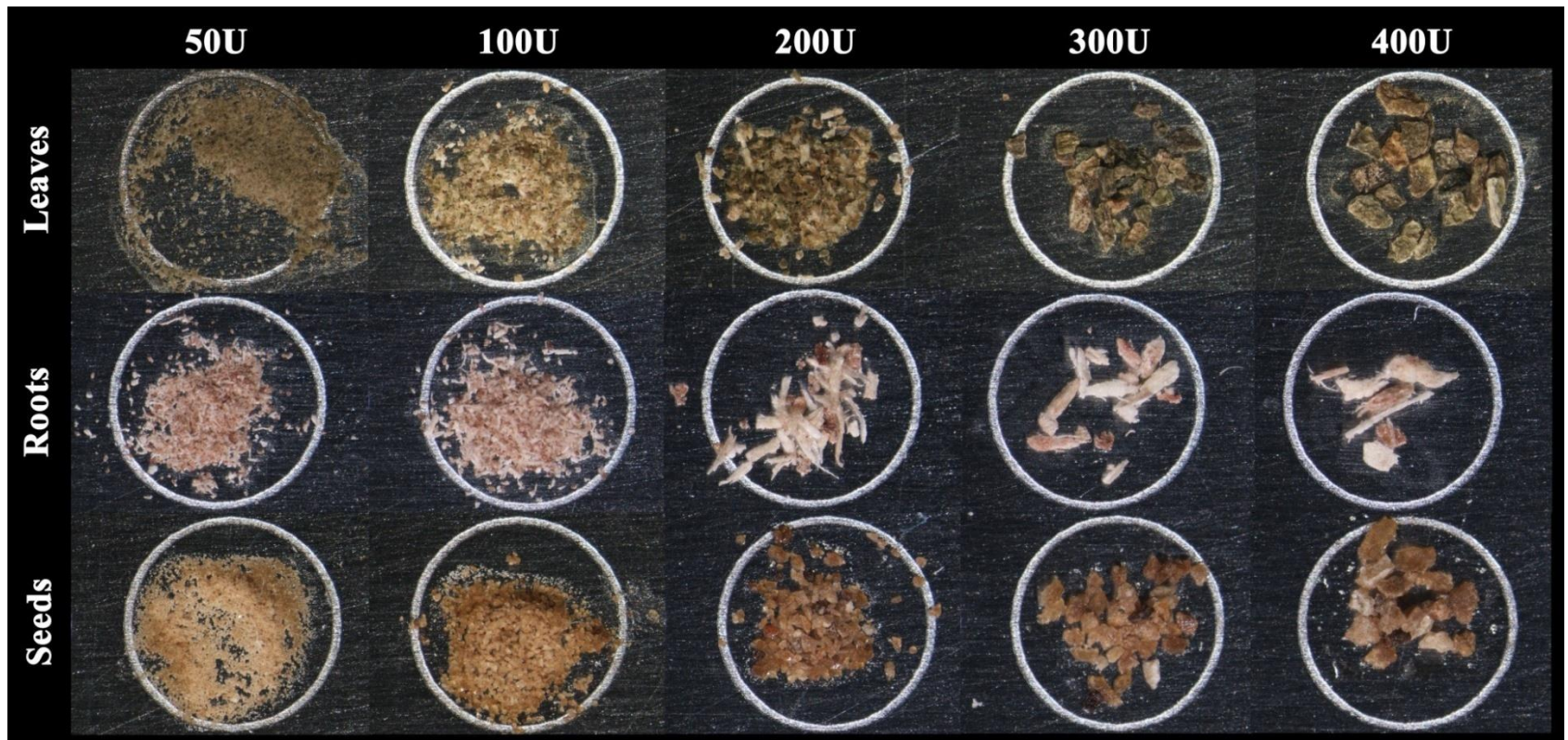
# Effect of Plant Parts: SEM Analysis



**Figure 3.** SEM Images of  $\leq 50 \mu\text{m}$  mesh particles of various parts of *Withania somnifera* treated with  $\text{CHCl}_3$  solvent.

**Musharraf et al, Journal of American Chemical Society of Mass Spectrometry, (25 (4), 530-537, 2014)**

# Effect of Plant Particle Size



**Figure 7.** Microscopic images of MALDI plate spotted with *Withania somnifera* plant material after passing through sieves of different mesh sizes.

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# Screening of various Plants Species

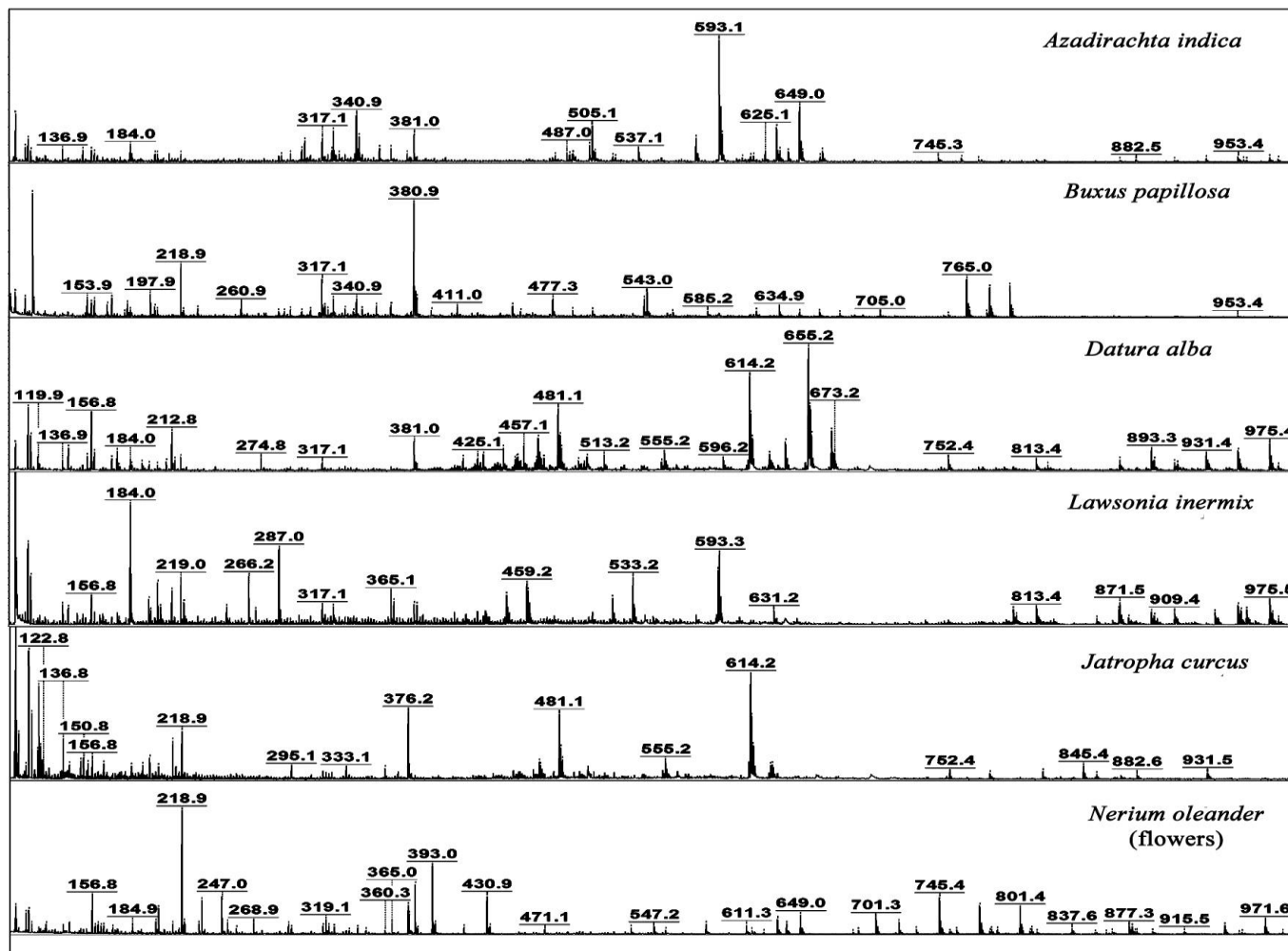


Fig.: TOF-MS Spectra of selected plants screened after treatment with different solvents.

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# Screening of various Plants Species

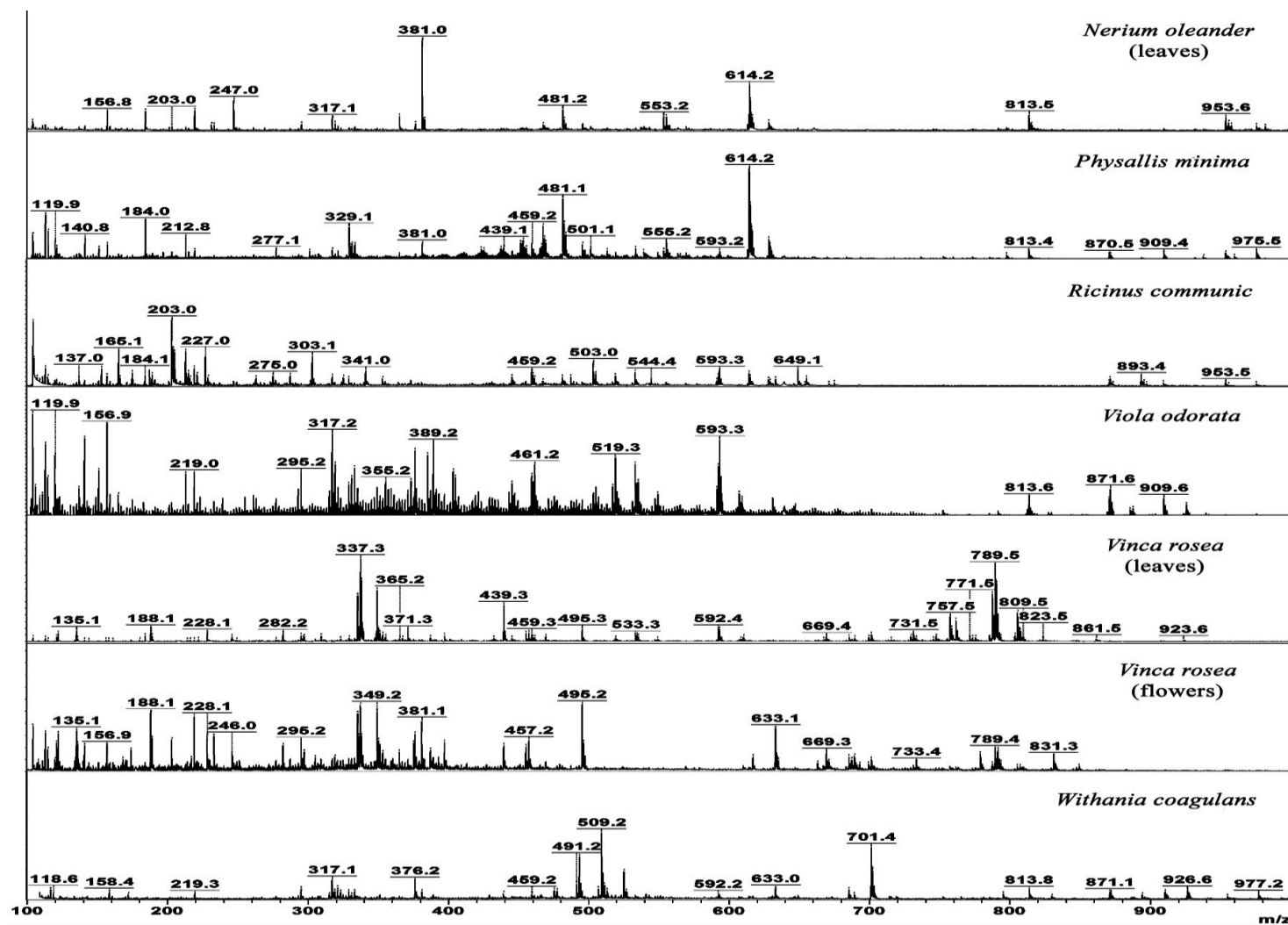


Fig: TOF-MS Spectra of selected plants screened after treatment with different solvents.

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# Characterization of Plant Metabolites

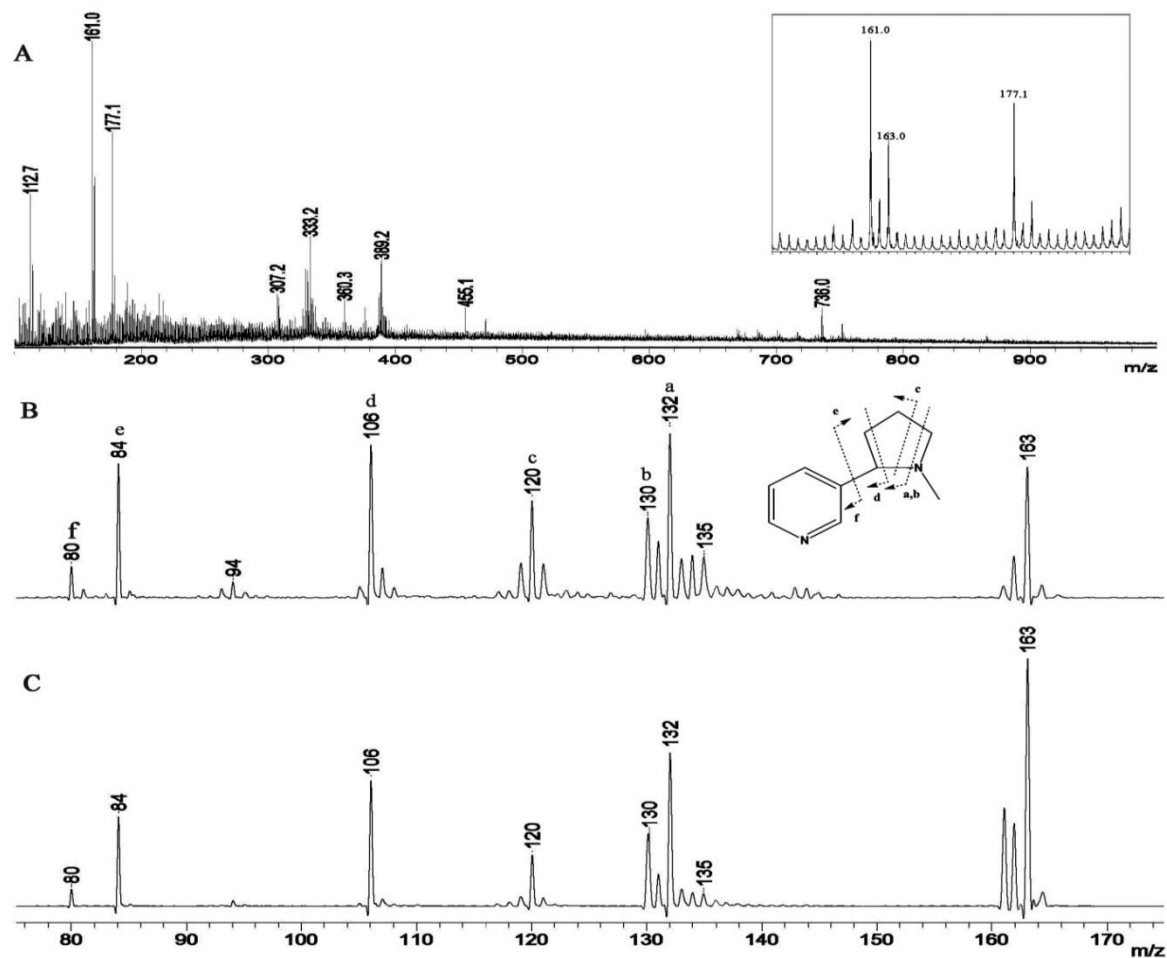


Fig. A) MALDI-TOF-MS spectrum of *Nicotiana tabacum* leaves powder.

B) Product ion spectrum of ion at  $m/z$  163. C) Product ion spectrum of nicotine standard solution.

# Characterization of Plant Metabolites in various Plants Species

No.	Observed <i>m/z</i>	Adduct ion	Plant analyzed	Proposed Metabolite
1	163	H <sup>+</sup>	<i>Nicotiana tabacum</i>	Cotinine
2	177	H <sup>+</sup>	<i>Nicotiana tabacum</i>	Nicotine
3	493	Na <sup>+</sup>	<i>Withania coagulans</i>	Coagulin R/ Coagulin J
4	463	H <sup>+</sup>	<i>Physallis minima</i>	Withaphysalin A/ Withaphysalin D
5	165	H <sup>+</sup>	<i>Ricinus communis</i>	Recinine
6	303	H <sup>+</sup>	<i>Ricinus communis</i>	19-Hydroxy-3,7,11-casbatrien-5-one
7	339	H <sup>+</sup>	<i>Catharanthus roseus</i>	Perivine/ Cathranthine/ Vindoline,
8	349	H <sup>+</sup>	<i>Catharanthus roseus</i>	Alstonine
9	809	H <sup>+</sup>	<i>Catharanthus roseus</i>	Vincathicine/ Leurosine
10	393	Na <sup>+</sup>	<i>Nerium oleander</i>	$\Delta^{16}$ -Dehydroadynenerigenin
11	553	Na <sup>+</sup>	<i>Nerium oleander</i>	Cardenolide N3
12	615	Na <sup>+</sup>	<i>Nerium oleander</i>	Neritaloside
13	694	Li <sup>+</sup>	<i>Nerium oleander</i>	Oleaside D
14	745	Li <sup>+</sup>	<i>Nerium oleander</i>	Glucosylnerigoside,
15	474	Li <sup>+</sup>	<i>Datura alba</i>	Withametelin C
16	655	Na <sup>+</sup>	<i>Datura alba</i>	Daturametelin J
17	673	Na <sup>+</sup>	<i>Datura alba</i>	Withametelin P
18	677	Na <sup>+</sup>	<i>Datura alba</i>	Baimantuoluoside A/B
19	365	Li <sup>+</sup>	<i>Lawsonia inermis</i>	Laxanthone II
20	543	Na <sup>+</sup>	<i>Buxus papilosa</i>	Buxanoldine

  **Laser Desorption Ionization Method for Plant powder analysis**

**LC-ESI-MS/MS analysis**

**Quantitative Analysis based on LC-MS/MS analysis**

# Mass Spectrometric Research Group

