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Bioinsecticidal Compounds of Celastraceae-the Spindle Tree Family

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Abstract: Celastraceae, family constitutes approximately 88 genera and 1300 species of plants. Celastraceae produce various β-dihydroagarofuran sesquiterpene polyesters and pyridine alkaloids, some of which exhibit insect antifeedant and insecticidal activities. Sesquiterpenes with α±-dihydro-β-agarofuran skeleton are considered to be chemotaxonomic indicators of the Celastraceae family. Most of the reported compounds are found to possess insecticidal activity.

Key words: Celastraceae, biopesticide, dihydroagarofuron, spindle tree

INTRODUCTION

Pesticides have done much to improve yields of high-quality agricultural products. But the long-term use of synthetic pesticides has many drawbacks, including toxicity to non-target organisms, development of pesticide resistance and environmental pollution. Renewed interest has been shown in the development of alternative strategies, including the use of suitable types of natural pesticides derived from a re-evaluation of age-old traditional botanical pest control agents. Insecticides derived from higher plants are a potential source of effective and safe pest control products. Clerodane diterpenes from various plant species (Belles *et al*., 1985) drimane type sesquiterpenes from *Warburgia* species and limonoids such as azadirachtin from Meliaceae are some of the best-studied natural products with insect antifeedant activity. Crude plant extracts of Celastraceae and many of the isolated products have been reported to possess antifeedant, insecticidal and insect repellent properties. Various species of Celastraceae have been used in Chinese traditional agriculture to protect crops from insect attack (Swingle *et al*., 1941). Plants of Celastraceae produce various β-dihydroagarofuran sesquiterpene polyesters and pyridine alkaloids, some of which exhibit insect antifeedant and insecticidal activities. Sesquiterpenes with α-dihydro-β-agarofuran skeleton are considered to be chemotaxonomic indicators of the Celastraceae family (Bruning and Wagner, 1978).

Celastraceae, the staff-tree family constitutes approximately 88 genera and 1300 species of plants (Mabberley, 1997). The family is principally a pantropical family of herbs, woody lianas, shrubs and trees. It is distributed widely throughout the world except for arctic regions. They have been valued since antiquity because their extracts have useful medicinal and insecticidal properties.

Over the last 30 years, a large number of secondary metabolites exhibiting a wide range of bioactivity have been extracted from the Celastraceae. The most pervasive and characteristic secondary metabolites isolated from this class of plants are large family of unusually highly oxygenated sesquiterpenoids having dihydro-β-agarofuran skeleton. Triterpenoids occurring in Celastraceae belongs predominantly to the friedelane (including quinone methide, ene-quinone methide and phenolic types), lupane and oleane series (Bruning and Wagner, 1978).

A significant chemotaxonomic characteristic of this family is the presence of quinine methide triterpenoids. Especially, euonyminol, 4β-hydroxyalatol and 1,4-deoxyalatol exhibits significant biological activities like anti-tumour, immunosuppressive, insecticidal, insect antifeedant and anti-HIV (Duan *et al*., 2000).

This review article was compiled to bring the highly potent insecticides and pesticides of Celastraceae to limelight.

INSECTICIDAL AND INSECT ANTI-FEEDANT ACTIVITY

Many species of Celastraceae family such as *Celastrus angulatus, C. orbiculatus, Tripterygium wilfordii T. foresstii* and *T. hypoglaucum*, are widely distributed and are used in traditional Chinese medicine for cancer treatment and as insecticides in china (Swingle *et al*., 1941; Jacobsen, 1958; Gonzalez *et al*., 1997). The plants produce various β-dihydroagarofuran sesquiterpene polyesters and pyridine alkaloids, some of

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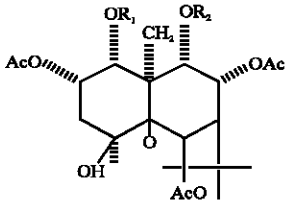
which exhibit insect antifeedant, insecticidal (Bruning and Wagner, 1978; Yamada *et al*., 1978; Tu, 1991).

Celastrus: Most of the species belonging to the genera *Celastrus* were reported to posses antifeedant and insectidical properties. The *C. angulatus* is commonly called as Chinese bittersweet, a woody climber distributed in china. The extracts are used as anti-feedant and it also has significant insecticidal properties. Traditionally, the powdered root bark of *C. angulatus* has been used in china to protect plant from insect damage. Investigations of the powdered root bark have demonstrated activity against several insect species. Included are cucumber beetle, *Aulacophora femoralis chinensis*, cruciferous leaf beetle, *Colaphellus bowringi*, willow leaf beetle, *Plagiodera versicolora*, cabbage sawfly, *Athalia flacca*, Hawaiian beet webworm, *Hymenia recurvalis*, imported cabbage worm, *Pieris rapae*, a tent caterpillar, *Malacosoma neustria testacea* and migratory locusts, *Locusta migratoria migratorioides* and *Locusta migratoria manilensis.* The biological activities of the extracts are reported to be due to the presence of angulatinA, Celangulins and sesquiterpenes.

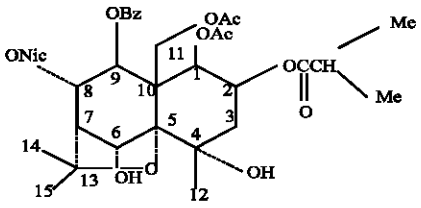
An insect anti-feedant celangulin, a sesquiterpenoids compound (non-alkaloidal) with dihydroagarofuran skeleton was isolated following fall worm army bioassay (*Spodoptera frugiperda*), from Chinese bittersweet *C. angulatus* (Wakabayashi *et al*., 1998). Celangulin [1] is thought to be the most active principle in the root bark and seed oil (Chiu, 1989).



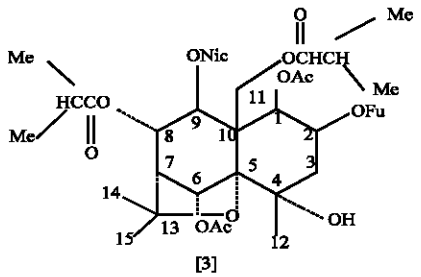
Celangulin I = R1 =Ac, R2 = z
2 R2 = z, R2 = Ac
[1]

Ji-Kai *et al*. (1990) isolated and tested about seven weakly basic alkaloids from *Celastrus angulatus* against *Pieris rapae*, *Ostrina furnacalis* and *Tribolium castaneum.* They found that these compounds (222 ppm) are strong antifeedants. It was observed that the insects were paralysed for many hours after ingesting the small dose of the test sample. The insects then recovered, fed and became paralysed again. As a result, the insects gradually starved to death. They reported the structure of four sesquiterpene alkaloids showing strong antifeeding action isolated from the root bark.

1β-acetoaxy isobutryloxy - 4α-hydroxy - 6α-hydroxy - 8α-nicotynoyloxy -9β-benzoyloxy-11α-acetoxy dihydro-β-agarofuran [2], 1β-acetoxy-2β-fungloxy-4α-hydroxy- 6α-acetoxy- 8β -isobutyryloxy- 9β -nicotynoyloxy-11α-isobutyryldihydro-β-agarofuran [3], isolated from *Celastrus angulatus* was reported to show strong antifeedant activity against several insects (Kai *et al*., 1991).

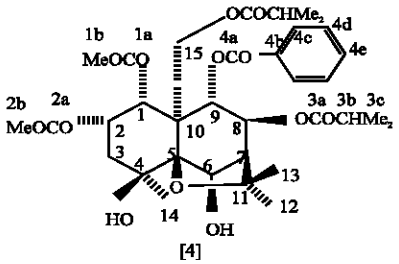


[2]



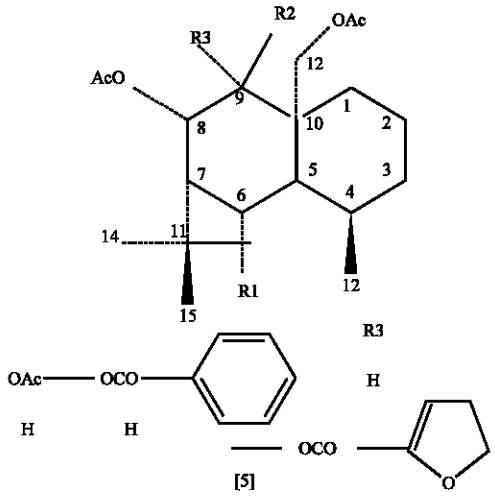
[3]

Maotian *et al*. (1991) isolated insecticidal sesquiterpene polyester, angulatin A [4] from the root bark of *C. angulatus*. The results of the pre-insecticidal tests showed that the sesquiterpene alkaloid mixture (25-50 ppm) containing angulatin A showed insecticidal and antifeeding effects against *Aphis gossypii* and *Pieris rapae*.



[4]

Hong *et al*. (1991) reported two new β-dihydroagarofuran sesquiterpene polyesters1-eq, 6-eq, 8-eq, 12-tetraacetoxy-9-eq-benzoxy-β-dihydroagarofuran [5] and 1-eq,8-eq,12-triacetoxy-9-ax-furaneancarboxy-β-dihydroagarofuran from *Celastrus paniculatus*.

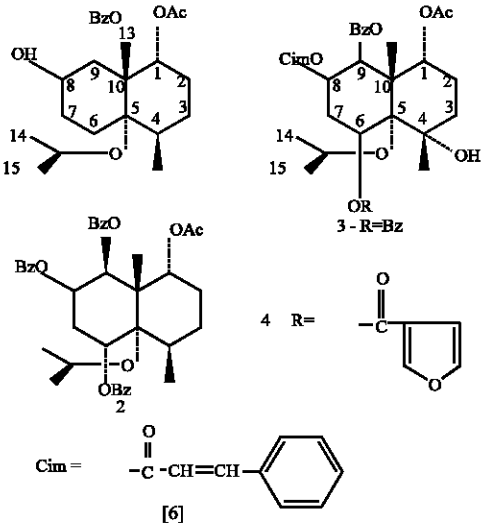


[5]

Tu (1991), elucidated structure of two new sesquiterpenoid insect antifeedants isolated from *Celastrus rosthorianus*. Dagang *et al.* (1992) isolated two sesquiterpenes, angulatueoid G and H from the seeds of *Celastrus angulatus* among which angulatueoid G (100 ppm) exhibited insect anti-feedant activity against *Aulacophora femoralis* (Antifeedant rate-73.2%) and *Piutella xylostella* (antifeedant rate-87.7%).

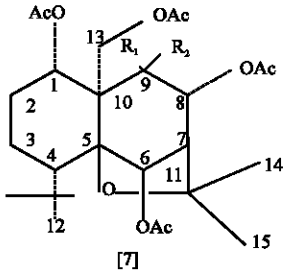
Wu *et al.* (1992), reported three insecticidal sesquiterpene polyol esters namely 1β,2β,6α-triaacetoxy-12-isobutanoyloxy-8β,9α-di(β-furancarbonyIoxy)-4α-hydroxy-β-dihydroagarofuran (celangulin II), 1β,2β,6α,8β-tetraacetoxy-9α-benzoyloxy-12-isobutanoyloxy-4α-hydroxy-β-dihydroagarofuran] (celangulin III) and 1β,2β, 6α,8β-tetraacetoxy-9β-benzoyloxy-12-isobutanoyloxy-4α-hydroxy-β-dihydroagarofuran (celangulin IV) from the root bark of *Celastrus angulatus*.

β-dihydroagarofuran sesquiterpenoids [6] were also isolated from the seed oil of *Celastrus paniculatus* (Tu and Chen, 1993).

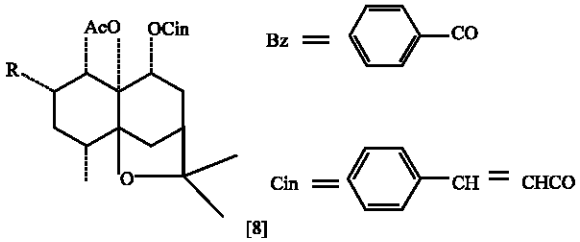


[6]

Wang and Chen (1997) isolated and elucidated the structure of new sesquiterpene polyol esters named celastrines A [7] and B [8], from *Celastrus falgellaris*, among which Celastrine A (500 mg L^{-1}) exhibited antifeedant activity (93%) against *Mythima upine*.



[7]



[8]

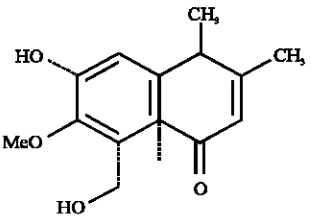
Wu *et al.* (2001), isolated five sesquiterpene polyol esters from the root bark of *Celastrus angulatus* by bioassay-guided fractionation. Their structures were determined by spectral data interpretation as 1α,2α,6β,8β,13-pentaacetoxy-9β-benzoyloxy-4β-hydroxy-β-dihydroagarofuran,1α,2α,6β-triacetoxy-8α-(β-furancarbonyloxy)-9β-benzoyloxy-13-isobutanoyloxy-4β-hydroxy-β-dihydroagarofuran, 1α,2α,6β-triacetoxy-8β-isobutanoyloxy-9β-(β-furancarbonyloxy)-13-(α-methyl) butanoyloxy-4β-hydroxy-β-dihydroagarofuran, 1α,2α,6β-triacetoxy-8α, 13-diisobutanoyloxy-9β-benzoyloxy-4β-hydroxy-β-dihydroagarofuran and 1α,2α,6β-triacetoxy-8α-isobutanoyloxy-9β-benzoyloxy-13-(α-methyl) butanoyloxy-4β-hydroxy-β-dihydroagarofuran which exhibited insecticidal activity against the larval of *Mythimna upine*.

Mingan *et al.* (2006), reported two insecticidal sesquiterpene polyol esters with a β-dihydroagarofuran sesquiterpene skeleton, celangulatin A, B, from *Celastrus angulatus*. The compounds exhibited insecticidal activities against *Mythimna upine*, which were demonstrated with the KD50 value of 68.5 and 215.8 μg L^{-1}, respectively.

Salacia: *Salacia* sp. Is known to possess elaborate anthocyanidines, catechins, phenolic acids, upine, friedooleanes, quinonemethie and related triterpenoids (Celastroids), mangiferin, gutta-percha and dulcitol (Bruning and Wagner, 1978; Sneden, 1981).

Two upine triterpenes, salacianone and salacinol have been isolated from the hexane extract of the stem bark of *Salacia beddomei* together with already reported compounds lup-20(29)-en-3-one, friedelan-3-one, 15α-hydroxyfriedelan-3-one, 15α-hydroxyfriedelane-1,3-dione, pristimerin and β-sitosterol (Hisham *et al*., 1995).

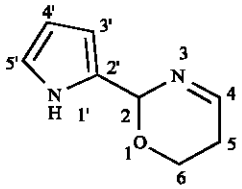
In Deepa *et al.* (2003) reported a new compound possessing benzoid skeleton [9] isolated from the leaves of *Salacia beddomei.* The antifeedant activity of the compound was tested against the cotton leaf worm *Spodoptera litura.* The compound at 0.5% (64%) and 1% (83%) concentration showed maximum antifeedant activity against the IV instar larvae of *S. litura* and 70% mortality was recorded with 1% extracts.



6-hydroxy-8-hydroxtmethyl-7methoxy-3, 4-dimethyl -4H-naphthalen01-one

[9]

Deepa *et al.* (2004), reported an oxaza cyclohexane [10] derivative from the nutrient stress stem callus of *Salacia beddomei*, which exhibited a significant antifeedant and insecticidal activity with 100% mortality on *Spodoptera litura.*

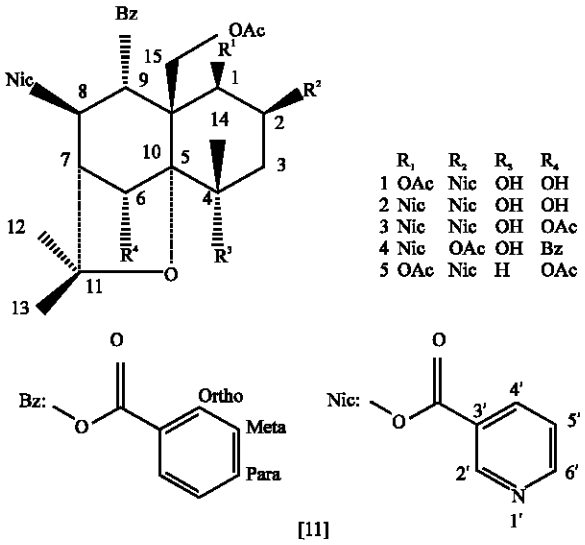


2-(21-pyroly1)-1,3-oxaza cyclohexane

[10]

Maytenus: Gonzalez *et al.* (1989) reported two new sesquiterpene polyesters with new polyhydroxy skeleton namely 1α, 9α-dibenzoyloxy-6β, 8α,15-triacetoxy-4β-hydroxy-dihydro-β-agarofruan and 1α,9α-dibenzoyloxy-2α, 6β, 8α,15-tetraacetoxy-4β-hydroxydihydro-β-agarofuran, from the aerial part of *Maytenus canariensis.* Gonzalez *et al.* (1990) isolated two poly esterified sesquiterpenes with dihydro- β-furan skeleton from *Maytenus canariensis.*

Itokawa *et al.* (1994) investigated the isolation and structural elucidation of 5 oligo-nicotinated sesquiterpene polyesters with a dihydroagarofuran core named cangorins F, G, H, I and J [11] from *Maytenus ilicifola.*

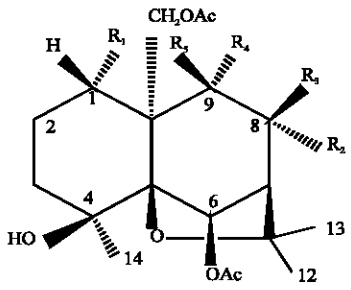


	R_1	R_2	R_3	R_4
1	OAc	Nic	OH	OH
2	Nic	Nic	OH	OH
3	Nic	Nic	OH	OAc
4	Nic	OAc	OH	Bz
5	OAc	Nic	H	OAc

[11]

Antifeedant and insecticidal activity of forty-one sesquiterpene with dihydroagarofuran skeleton and 14 related synthetic compounds were assayed for antifeedant and insecticidal activities against Egyptian cotton leaf worm *Spodoptera littoralis* using leaf disk method. Of the sesquiterpenes assayed 38 showed antifeedant activity and three compounds demonstrated insecticidal activity (Gonzalez *et al*., 1997).

Phytochemical study of the leaves of *Maytenus macrocarpa* revealed fewer sesquiterpene than other *Maytenus* species with triterpene being the main secondary metabolite. The aerial parts of *Maytenus macrocarpa* yielded three new β-dihydroagarofuran sesquiterpene polyol esters, 6β,8β-15-triacetoxy-1α,9α-dibenzoyloxy-4β-hydroxy-β-dihydroagarofuran, 1α, 6β, 8β, 15-tetraacetoxy-9α-benzoyloxy-4β-hydroxy-β-dihydroagarofuran and (1S, 4S, 6R, 7R, 8R, 9R)-1, 6, 15-triacetoxy-8, 9-dibenzoyloxy-4-hydroxy-β-dihydroagarofuran [12] (Chavez *et al*., 1999).



$R_1 = R_3$ - Obz; $R_2 = R_4 = H$; $R_5 =$ Oac
$R_1 = R_3 =$ Oac; R_4-Obz; $R_2 = R_5 = H$
$R_1 =$ Oac; $R_2 = R_5 =$ Obz; $R_3 = R_4 = H$

[12]

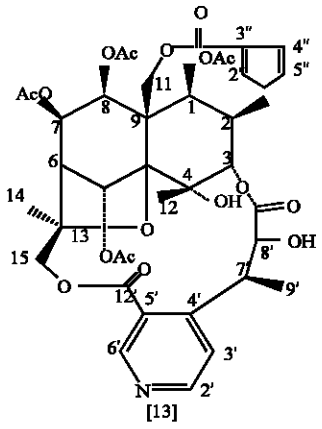
Avilla *et al.* (2000), isolated nortriterpene quinone methides (pristimerin, tingenonee and

20-α-hydroxytingenone), from *Maytenus* sp which showed the same effect like that of azadirachtin on codling moth (*Cydia pomonella*, Lepidoptera: Tortricidae). Among these three compounds 20-α-hydroxytingenone was reported to be highly active compound, showing lethal, antifeedant and insect growth regulation activities. Pristimerin showed also a high antifeedant activity together with its molt effect suppression. Tingenone showed the lowest activity. The differences in the activity of the three products are related to the structure of the E ring.

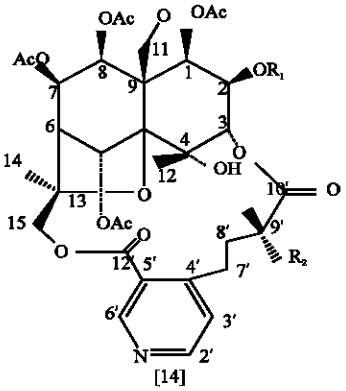
From the aerial parts of *Maytenus disticha*, 9β-benzoyloxy-1α,2α,6β,8α,15-penta -acetoxy - dihydro-β-agarofuran and from seeds of *Maytenus boaria* 9β-furoyloxy-1α,6β,8α-triacetoxy-dihydro-β-agarofuran were isolated. The compounds tested for activity using neonate larvae in a no choice artificial diet bioassays, the agarofurans and the MeOH and hexane/EtOAc extracts caused significant growth inhibitory effects comparative to toosendanin (a compound from *Melia azadirach).* Compounds caused 100% larval mortality at 25 and 15 ppm, respectively. MeOH and hexane/EtOAc extracts caused 100% larval mortality at 25.0 ppm, respectively, they also increased the development time of surviving larvae and a significant delay for the time of pupation and adult emergence. These compounds showed comparable potency of activity with toosendanin. Acute toxicity against adults of *S. frugiperda* was also found, for hexane/EtOAc extract and 9β-furoyloxy-1α,6β,8α-triacetoxy-dihydro-β-agarofuran had the most potent activity with LD50 value of 4.7 and 1.9 ppm, respectively. MeOH extract, hexane/EtOAc extract, both the compounds caused acetylcholinesterase inhibition with 78.0, 89.2, 79.3 and 100% inhibition at 15.0 ppm, respectively. Thus it was concluded that furoyloxy agarofuran might be responsible for the insecticidal activity of these plants (Cespedesa *et al*., 2001). Pristimerin isolated from *Maytenus illicifolia* has been shown to be cytotoxic to several cancer cell lines. Pristimerin reduced the number of viable cells and increased number of non-viable cells. The study showed the importance of pristimerin as representative of an emerging class of potential anticancer chemicals, exhibiting an antiproliferative effect by inhibiting DNA synthesis and triggering apoptosis (Costa *et al*., 2008).

15α-Hydroxy-21-keto-pristimerine, a new nortriterpene quinone methide was isolated from the root bark of *Maytenus catingarum* along with other well-known related compounds, including pristimerine, tingenone and 20α-hydroxy-tingenone (Alvarenga *et al*., 1999).

Tripterygium: The first insecticidal constituents were isolated from the extracts of *Tripterygium wilfordii* and characterized as the sesquiterpenoids wilfordine and wilforine. These alkaloids, which are based on the euonyminol core, were found to be highly toxic towards the larvae of the European corn borer *Pyrausta nubialis* at concentrations of 60 ppm (100% mortality after 3 days). Additionally wilforine [14] displays effective antifeedant activity towards cabbage leaf worm *Pieris rapae* at 1 X 10^{-5}% dry weight (Monache *et al*., 1984). Li *et al*. (1999), isolated Wilfordine from *Tripterygium hypoglaucum.* Li *et al*. (1997), isolated the terpenoids from *Tripterygium wilfordii.* From the roots of *Tripterygium hypoglaucum*, a novel sesquiterpene pyridine alkaloid Hypoglaunine [13] was isolated along with other known compounds (Li *et al*., 1999).



[13]



[14]

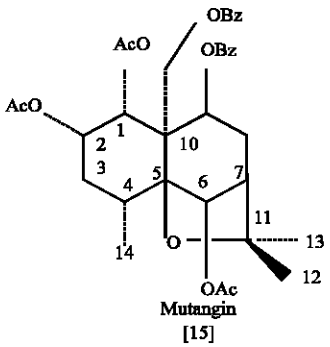
Hypoglaunine		R1		R2
	2	C6H5CO	OH	Wilfordine
	3	(C4H3O)CO	OH	Wilformine
	4	CH3CO	H	Wilformine
	5	(C5H5N)CO	H	Wilfornine

Euonymus: Various β-dihydroagarofurnan sesquiterpene polyesters and pyridine alkaloids were reported from the

plants of the genus *Euonymus* (Bruning and Wagner, 1978; Yamada *et al*., 1977, 1978, Ishiwata *et al*., 1983; Rozsa and Pelczer, 1989; Rozsa *et al*., 1989; Han *et al*., 1990a,b; Tu, 1991; Hohmann, 1995; Wang *et al*., 2000). Jinbo *et al*. (2002) isolated the three sesquiterpene pyridine alkaloids with β-dihydroagarofuran skeleton namely euoverrine A, B and euphelline along with the already reported euojaponine, from the root bark of *Euonymus verrucosides, E. fortunii* ad *E. phellomana* by bioassay guided fractions.

Other genera: Gonzalez *et al*. (1987) isolated sesquiterpene alkaloids with dihydroagarofuran ring from the ariel parts of *Orthosphenia exicana* and root bark of *Rzedowskia tolantonguensis*. 1α-benzoyloxy-6β-nicotinoyloxy-9β-acetoxy-4β-hydroxydihydro-β-agarofuran and 1α-cinnamoyloxy-9a-acetoxy-4b-8b-dihydroxydihydro-b-agarofuran was isolated from *Orthosphenia exicana*. They also reported (1) and 1α-benzoyloxy-6β-nicotinoyloxy-8β-9a-diacetoxy-4β-hydroxydihydro-β-agarofuran from *Rzedowskia tolantonguensis*.

The antifeedant activity of 15 polyesterified sesquiterpenes from Celastraceae against the Egyptian cotton leaf worm *Spodoptera littoralis* was also reported. (Gonzalez *et al*., 1992). Tsanuo *et al*. (1993) isolated a novel sesquiterpene Mutangin [15] with moderate antifeedant activity against the lepidopteran, *Chilo pratellus* from unripe fruits of *Elaeodenron buchnanii*.



Mutangin [15]

CONCLUSION

Review on insecticidal and antifeedant properties of the crude extracts or isolated compounds of members of Celastraceae will open up a new avenue for applying these plants in crop protection programmes. There are many unexploited members belonging to the family Celastraceae are distributed in India. These plants should be taken for research and the applications and the use of these plants as potential insecticides should be explored. Further research should be carried out on commercialization of these useful plants of Celastraceae as biocontrol agents.

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