



PHYTOCHEMISTRY OF *CLERODENDRUM SERRATUM* (L.) MOON.: A REVIEW

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ABSTRACT

Phytochemical study of natural compounds has recently undergone exponential growth due to advances in isolation techniques, spectral characterization and biological evaluation. *Clerodendrum serratum* (L.) Moon. has been studied extensively for presence of phytochemicals by number of recent scientific literature. The phytochemical review on *C. serratum* has compiled from electronic databases, official and non-official reference books, scientific journals, periodicals and SCOPUS, Google Scholar, NOPR, PubMed, Springer, Elsevier, ACS, Medline Plus and Web of Science. Literature on phytochemical study of *C. serratum* revealed more than 35 secondary metabolites consisting of different chemical classes of compounds. Modern instrumental techniques of UV, IR, NMR and mass spectrometry have been utilized to illustrate structure of compounds. The compounds like terpenoids, steroids, iridoids, phenyl propanoids, flavonoids and carbohydrates has been identified from bark, root, stem, leaves and aerial parts. *C. serratum* possesses pharmacological activities like anti-inflammatory, antioxidant, anti-asthmatic, anticancer, hepatoprotective and antibacterial is supported by literature.

Key words: *Clerodendrum serratum*; Phytochemistry; Terpenoids; Flavanoids; Sterols.

INTRODUCTION

The *Clerodendrum* genus is very huge member of Verbenaceae family. It contains about 450 species which is widely scattered in tropical and warm temperate regions of Asia, Australia, Africa and America (Mabberley *et al.*, 2008). This review is focused on phytoconstituents of *Clerodendrum serratum* (L.) Moon. The phytochemical studies resulted in the isolation of more than 35 compounds consisting of different chemical classes of compounds dominated by terpenoids (Banerjee *et al.*, 1969; Rangaswami and Sarangan, 1969; Fan *et al.*, 2007; Ganapaty *et al.*, 1997; Vidya *et al.*, 2007; Bhujbal *et al.*, 2010a; Boonsri, 2004; Juvekar *et al.*, 2006), sterols

(Ganapaty *et al.*, 1997; Banerjee *et al.*, 1969; Boonsri, 2004; Nair *et al.*, 1976; Fan *et al.*, 2007), iridoids (Wei *et al.*, 2000 ; Yang *et al.*, 2000b), phenyl propanoids (Nair *et al.*, 1976; Yang *et al.*, 2000a; Fan *et al.*, 2007; Wei *et al.*, 2000), flavonoids (Bhujbal *et al.*, 2010a; Nair *et al.*, 1976; Ganapaty *et al.*, 1997; Fan *et al.*, 2007) and carbohydrates (Garg and Verma, 1966; Boonsri, 2004; Juvekar *et al.*, 2006) present in bark, root, stem, aerial parts and leaves.

To date many research reports and limited number of reviews published recently deals about clinical uses, traditional uses and pharmacological activities of *C. serratum* (Patel *et al.*, 2014; Praveen Kumar and Nishitewar, 2013; Singh *et al.*, 2012; Srivastava and Patel, 2007). The aim of the present review was to deliver spectral data of major natural compounds present in *C. serratum*. The information provided in this review is

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compiled by using electronic search (using SCOPUS, Google Scholar, NOPR, PubMed, Springer, Elsevier, ACS, Medline Plus and Web of Science) and library search. In this review special emphasis is given to the documented natural compounds, chemical composition of different plant parts and spectral characterization of major phytoconstituents.

Phytochemistry

Plants can produce different types of secondary metabolites, which have been subsequently utilized by humans for their valuable characters in a diverse array of applications (Zwenger and Basu, 2008). Secondary metabolites include compounds produced in response to stress, such as the case when acting as a deterrent against herbivores (Keeling *et al.*, 2006). Many authors attempted to isolate secondary metabolites from different parts of the *Clerodendrum serratum* as shown in Table 1 and their structures are presented in figure 1. The major chemical constituents are terpenoids, saponins, phenolics, flavonoids and carbohydrates. These metabolites and their biological effects have been thoroughly covered in number of reports (Patel *et al.*, 2014; Praveen Kumar and Nishteswar, 2013; Singh *et al.*, 2012; Shrivastava and Patel, 2007).

Clerodendrum serratum possesses different pharmacological activities like antiviral, antibacterial, antimalarial, anti-inflammatory, inhibition of cholesterol synthesis and anticancer activities. Oleanolic acid, documented for its anti-inflammatory and cytotoxic effects, has been reported as the major constituent of the triterpenoid portion of the drug (Mann *et al.*, 1994; Liu, 1995). Oleanolic acid, queretaroic acid and serratagenic acid were reported from root of the plant (Juvekar *et al.*, 2006). Sterols like β -sitosterol, γ -sitosterol, spinasterol, spinasteryl- β -D-glucopyranoside, α -spinasterol, stigmasterol and Bis(2-ethylhexyl) phthalate and Serratum A are found to be present in the stem, leaves, root and aerial parts. Several ostersterols isolated from plants are listed in Table 1 (Shrivastava and Patel, 2007). Two iridoid glucosides, serratoside A and serratoside B, were reported from the aerial parts of *Clerodendrum serratum*. 7- β -coumaroyl oxyugandoside and 7- β -cinnamoyl oxyugandoside were also isolated from leaves of the plant (Wei *et al.*, 2000a).

In the *Clerodendrum* genus phenolics are reported to be present in both free as well as bound to sugar moieties (Harbone, 1984; Mann *et al.*, 1994). From roots and leaves of the plant phenolic compounds have been separated (Table 1). Phenyl propanoids like serratumoside-A, martynoside, myricoside and aceteoside have been isolated from the aerial parts of *C. serratum* (Yang *et al.*, 2000a; Fan *et al.*, 2007). Flavonoids are secondary metabolites characterised by flavan nucleus and C6-C8-C6 carbon-skeleton (Peterson and Dwyer,

1998; Tsuchiya, 2010). These are group of structurally related compounds with a chromane-type skeleton having phenyl substituent in C2-C3 position (Rijke *et al.*, 2006). The basic structural feature of flavonoid is 2-phenylbenzo- γ -pyrane nucleus consisting of two benzene rings linked through a heterocyclic pyran ring. Free as well as bound flavonoid aglycones are present in different forms like catechins, flavanones, flavanols, flavones, flavonols, chalcones, aurones and isoflavones (Harbone, 1984; Mann *et al.*, 1994). They are reported to display potent antioxidant, antimicrobial, antiasthmatic, cytotoxic and central nervous system activities (Shrivastava and Patel, 2007). Apigenin and luteolin glucosides have been reported from stem and leaves while a very uncommon and rare, 6-hydroxyluteolin was reportedly found present in leaves.

Oleanolic acid (Gohari *et al.*, 2009; Kovač-Bešović *et al.*, 2009)

Molecular Formula: C₃₀H₄₈O₃

UV (λ_{max} , EtOH nm): 277.

IR (KBr) cm⁻¹: 3426 (-OH), 2864 (-CH₂), 1698 (C=O), 1462 (-OH), 1376 (-CH₃), 1108 (C-O).

¹H-NMR (δ CDCl₃, 500 MHz): 0.75, 0.77, 0.90, 0.91, 0.93, 0.98 (each 3H, s, CH₃ \times 6), 1.13 (3H, s, H-27), 2.82 (1H, dd, J =3.6 Hz & 13.2 Hz, H-18), 3.23 (1H, dd, J =11.2 Hz & 4.4 Hz, H-3), 5.27 (1H, t, J =3.5 Hz, H-12).

¹³C-NMR (δ Pyridine-d5, 125 MHz): 39.0 (C-1), 28.2 (C-2), 78.1 (C-3), 39.4 (C-4), 55.8 (C-5), 18.8 (C-6), 33.3 (C-7), 39.8 (C-8), 48.2 (C-9), 37.4 (C-10), 23.7 (C-11), 122.6 (C-12), 144.8 (C-13), 42.2 (C-14), 28.4 (C-15), 23.8 (C-16), 46.7 (C-17), 42.0 (C-18), 46.5 (C-19), 31.0 (C-20), 34.3 (C-21), 33.2 (C-22), 28.8 (C-23), 16.6 (C-24), 15.6 (C-25), 17.5 (C-26), 26.2 (C-27), 180.2 (C-28), 33.3 (C-29), 23.8 (C-30).

MS (m/z): 320, 306, 279, 203, 289, 173, 159, 147, 129, 119, 105, 95, 81.

Serratagenic acid (Yu *et al.*, 1995)

Molecular Formula: C₃₀H₄₆O₄

IR (KBr) cm⁻¹: 3386, 2945, 2835, 1667, 1637, 1454.

¹H-NMR (δ , CD₃OD, 500MHz): 1.8 (m, H-1), 1.30 (m, H-1), 1.76 (m, H-2), 1.40 (m, H-2), 3.15 (dd, J =11.5 Hz & J =5.0 Hz H-3), 0.75 (m, H-5), 1.58 (m, H-6), 1.34 (m, H-6), 1.63 (m, H-7), 1.34 (m, H-7), 1.62 (m, H-9), 1.98 (m, H-11), 1.69 (m, H-11), 5.30 (br.t, J =3.5 Hz H-12), 1.59 (m, H-15), 1.11 (m, H-15), 1.64 (m, H-16), 1.32 (m, H-16), 2.70 (dd, J =14.0 Hz and J =4.0 Hz H-18), 1.92 (m, H-19), 1.64 (m, H-19), 1.95 (m, H-21), 1.66 (m, H-21), 1.90 (m, H-22), 1.63 (m, H-22), 0.97 (s, H-23), 0.80 (s, H-24), 0.94 (s, H-25), 0.77 (s, H-26), 1.16 (s, H-27), 1.13 (s, H-30).

¹³CMR (δ , CD₃OD, 125MHz): 38.6 (t, C-1), 26.6 (t, C-2), 78.5 (d, C-3), 39.3 (s, C-4), 55.5 (d, C-5), 18.3 (t, C-6), 32.8 (t, C-7), 39.3 (s, C-8), 48.3 (d, C-9), 36.9 (s, C-10),

23.0 (t, C-11), 123.0 (d, C-12), 143.5 (s, C-13), 41.5 (s, C-14), 27.6 (t, C-15), 23.3 (t, C-16), 45.8 (s, C-17), 42.8 (d, C-18), 42.1 (t, C-19), 43.8 (s, C-20), 30.1 (t, C-21), 33.8 (t, C-22), 27.4 (q, C-23), 15.1 (q, C-24), 14.7 (q, C-25), 16.4 (q, C-26), 25.1 (q, C-27), 180.0 (s, C-28), 177.6 (s, C-29), 27.5 (q, C-30).

MS: 470 [M+H]⁺

Ursolic acid (Suhagia *et al.*, 2013; Uddin *et al.*, 2011)

Molecular Formula: C₃₀H₄₈O₃

UV (λ_{max} , MeOH, nm): 212.4.

IR (KBr) cm⁻¹: 3427 (OH), 1689.53 (C=O), 2650, 2358.7.

¹HNMR (δ , 300MHz): 3.43 (br. s, H-3), 5.50 (br. s, H-12), 2.52 (d, J=11.0 Hz, H-18), 1.24 (s, H-23), 1.02 (s, H-24), 0.93 (s, H-25), 1.05 (s, H-26), 1.22 (s, H-27), 0.97 (s, H-29), 0.99 (s, H-30).

¹³CMR (δ , CDCl₃, 75MHz): 38.4 (C-1), 28.1 (C-2), 78.1 (C-3), 38.4 (C-4), 55.8 (C-5), 18.8 (C-6), 33.6 (C-7), 40.0 (C-8), 48.3 (C-9), 37.4 (C-10), 23.6 (C-11), 125.6 (C-12), 139.7 (C-13), 42.5 (C-14), 28.7 (C-15), 24.9 (C-16), 48.0 (C-17), 53.5 (C-18), 39.5 (C-19), 39.1 (C-20), 31.1 (C-21), 37.3 (C-22), 28.8 (C-23), 15.7 (C-24), 16.6 (C-25), 17.4 (C-26), 23.8 (C-27), 180.0 (C-28), 17.5 (C-29), 21.4 (C-30).

MS: 455 [M]⁺, 439, 248, 203, 189, 119.

Bauer-9-en-3-one (Boonsri, 2004)

Molecular Formula: C₃₀H₅₀O

IR (CHCl₃) cm⁻¹: 1709, 3300, 1408.

¹HNMR (δ CDCl₃): 2.09 (1H, ddd, J=13.5 Hz, 6.5 Hz, & 3.5Hz, H-1), 1.78 (1H, m, H-1), 2.72 (1H, ddd, J=15.5Hz, J=13.5 Hz, & 6.5 Hz, Ha-2), 2.40 (1H, ddd, J=15.5Hz, 5.5 Hz, & 3.5 Hz, Hb-2), 1.35 (1H, m, H-5), 1.46 (1H, m, H-6), 0.38 (1H, m, H-6), 1.23 (2H, m, H-7), 2.07 (1H, m, H-8), 5.29 (1H, d, J=6.5 Hz, H-11), 1.44 (1H, m, H-12), 1.26 (1H, m, H-12), 1.34 (1H, m, H-15), 1.30 (1H, m, H-15), 1.60 (2H, m, H-16), 1.60 (1H, m, H-18), 0.98 (1H, q, J=9.5 Hz, H-19), 1.86 (1H, m, H-21), 1.24 (1H, m, H-21), 1.70 (2H, m, H-22), 1.07 (3H, s, H-23), 1.07 (3H, s, H-24), 1.21 (3H, s, H-25), 0.79 (3H, s, H-26), 0.81 (3H, s, H-27), 0.77 (3H, s, H-28), 0.83 (3H, d, J=6.0 Hz, H-29), 0.89 (3H, d, J=6.5 Hz, H-30).

¹³CMR (CDCl₃): 36.64 (C-1), 34.89 (C-2), 217.30 (C-3), 47.64 (C-4), 53.26 (C-5), 35.86 (C-6), 28.18 (C-7), 41.0 4(C-8), 147.41 (C-9), 39.30 (C-10), 115.61 (C-11), 29.62 (C-12), 36.75 (C-13), 38.18 (C-14), 20.15 (C-15), 22.57 (C-16), 42.80 (C-17), 51.99 (C-18), 59.59 (C-19), 30.77 (C-20), 26.27 (C-21), 36.07 (C-22), 22.04 (C-23), 25.53 (C-24), 21.64 (C-25), 15.29 (C-26), 16.95 (C-27), 13.98 (C-28), 22.99 (C-29), 22.10 (C-30).

EI-MS (m/z): 426 [M]⁺

Serratin (Ravikumar *et al.*, 2008)

Molecular Formula: C₂₄H₄₀O₂

UV (λ_{max} , MeOH, nm): 214, 238.

IR (CHCl₃) cm⁻¹: 3400, 1712, 1635, 1600, 1360, 870.

¹HNMR (δ CDCl₃): 2.10 (2H, br. s, H-2), 2.20 (2H, br. s, H-4), 0.77, 0.98, 1.02 (each 3H, s, H-18, H-19, H-20), 4.61 and 4.78 (2H, br. s, =CH₂), 1.70 (3H, s, =C-CH₃), 5.35 and 5.45 (each 1H, m, -C=C-H).

¹³CMR (δ CDCl₃): 32.0 (C-1), 22.6 (C-2), 216.5 (C-3), 21.3 (C-4), 55.4 (C-5), 18.2 (C-6), 129.2 (C-7), 147.0 (C-8), 152 (C-9), 37.1 (C-10), 132.0 (C-11), 25.1 (C-12), 36.2 (C-13), 56.2 (C-14), 24.4 (C-15), 28.4 (C-16), 56.4 (C-17), 12.1 (C-18), 21.5 (C-19), 74.5 (C-20), 19.0 (C-21), 33.9 (C-22), 30.8 (C-23), 36.4 (C-24), 150.9 (C-25), 20.2 (C-26), 109.3 (C-27).

EI-MS (m/z): 396 (14), 378 (22), 326 (31), 309 (27), 269 (25), 286 (56), 272 (48), 222 (70), 168 (100), 174 (82), 124 (40), 110 (52), 70 (51), 41 (69).

Lupeol (Haque *et al.*, 2006; Ravikumar *et al.*, 2008)

Molecular Formula: C₃₀H₅₀O

UV (λ_{max} , nm): 350.

IR (KBr) cm⁻¹: 3610, 3070, 3015, 1640, 1520, 1380, 1217, 1020, 887.

¹HNMR (δ CDCl₃): 0.75, 0.78, 0.81, 0.92, 0.94, 1.02 (Me-28, Me-23, Me-24, Me-25, Me-26, Me-27), 1.67 (3H, br. d, J=0.5 Hz, Me-30), 3.18 (1H, dd, J=9.6 & 6.2 Hz, H_a-3), 4.56 (1H, d, J=0.4 Hz, Ha-29), 4.67 (1H, dq, J=0.4, 0.5 Hz, H_b-29)

¹³CMR (δ CDCl₃): 38.0 (C-1), 27.4 (C-2), 79.0 (C-3), 38.7 (C-4), 55.3 (C-5), 55.3 (C-5), 18.3 (C-5), 18.3 (C-6), 34.2 (C-7), 40.1 (C-8), 50.4 (C-9), 37.7 (C-10), 20.9 (C-11), 25.1 (C-12), 38.0 (C-13), 42.8 (C-14), 27.4 (C-15), 35.6 (C-16), 42.8 (C-17), 48.2 (C-17), 48.2 (C-18), 48.0 (C-19), 150.9 (C-20), 28.5 (C-21), 40.0 (C-22), 28.1 (C-23), 15.4 (C-24), 16.1 (C-25), 15.9 (C-26), 14.6 (C-27), 18.0 (C-28), 109.5 (C-29), 19.4 (C-30).

EI-MS (m/z, relative intensity): 426 [M]⁺ (2), 411 [M⁺ - CH₃] (3), 408 [M⁺ - H₂O] (3), 218 (5), 207 (6), 189 (58), 163 (80), 135 (57), 107 (68), 105 (55), 79 (54), 41 (100).

β -sitosterol (Chaturvedula and Prakash, 2012; Sen *et al.*, 2012)

Molecular Formula: C₂₉H₅₀O

UV (λ_{max} , nm): 208

IR (KBr) cm⁻¹: 3426.3, 2936, 2832, 2366, 1596.4.

¹HNMR (δ CDCl₃, 600 MHz): 3.53 (tdd, 1H, J = 4.5 Hz, 4.2 Hz & 3.8 Hz), 5.36 (t, 1H, J = 6.4 Hz) , 0.93 (d, 3H, J = 6.5 Hz), 0.84 (t, 3H, J = 7.2 Hz), 0.83 (d, 3H, J = 6.4 Hz), 0.81 (d, 3H, J = 6.4 Hz) , 0.68 (s, 3H), 1.01 (s, 3H).

¹³CMR (δ CDCl₃, 150 MHz): 37.5 (C-1), 31.9 (C-2), 72.0 (C-3), 42.5 (C-4), 140.9 (C-5), 121.9 (C-6), 32.1 (C-7), 32.1 (C-8), 50.3 (C-9), 36.7 (C-10), 21.3 (C-11), 39.9 (C-12), 42.6 (C-13), 56.9 (C-14), 26.3 (C-15), 28.5 (C-16), 56.3 (C-17), 36.3 (C-18), 19.2 (C-19), 34.2 (C-20), 26.3 (C-21), 46.1 (C-22), 23.3 (C-23), 12.2 (C-24), 29.4 (C-25), 20.1 (C-26), 19.6 (C-27), 19.0 (C-28) , 12.0 (C-29).

MS (m/z): 414[M⁺], 396, 339, 325, 310, 298, 257, 227, 140, 139, 125, 97, 71, 57.

γ -sitosterol (Jain *et al.*, 2009; Nyamoita *et al.*, 2013)Molecular Formula: C₂₉H₅₀OUV (λ_{max} , nm): 251IR (CHCl₃) cm⁻¹: 3319, 2946, 2854, 1640, 1470, 1189, 1060, 870, 720, 670.¹HNMR (δ CDCl₃): 5.10 (m, 1H, H-6), 3.51 (tdd, 1H, H-3), 1.26 (s, 3H), 1.17 (s, 3H), 1.00 (s, 3H), 0.91 (s, 3H), 0.90 (s, 3H).13CMR (δ CDCl₃): 37.0 (C-1), 29.5 (C-2), 71.8 (C-3), 42.3 (C-4), 140.8 (C-5), 121.7 (C-6), 31.9 (C-7), 29.2 (C-8), 50.2 (C-9), 36.5 (C-10), 21.1 (C-11), 26.1 (C-12), 45.9 (C-13), 56.7 (C-14), 24.1 (C-15), 39.8 (C-16), 56.1 (C-17), 12.2 (C-18), 18.8 (C-19), 34.0 (C-20), 19.1 (C-21), 37.3 (C-22), 26.6 (C-23), 50.1 (C-24), 28.3 (C-25), 19.4 (C-26), 19.8 (C-27), 23.3 (C-28), 12.0 (C-29).MS (m/z, %): 414 [M⁺], 43 (100), 396 (8), 381 (6), 329 (2), 303 (2), 275 (10), 255 (14), 213 (13), 199 (8), 159 (25), 147 (34), 145 (48), 131 (25), 121 (32), 107 (34), 105 (43), 81 (61), 69 (36), 57 (55), 55 (64).**Spinasterol** (Ragasa *et al.*, 2005; Billah *et al.*, 2013)Molecular Formula: C₂₉H₄₈OIR (KBr) cm⁻¹: 3456 (OH), 3050 (H-C=C), 2930, 3850, 1640, 1450, 1370, 1040, 970, 830.¹HNMR (δ CDCl₃, 400 MHz): 1.09, 1.82, 1.39, 1.77, 3.59, 1.27, 1.70, 1.40, 1.22, 1.74, 5.12 (br. s), 1.65, 1.48 (2H), 1.23, 2.02, 1.81, 1.40, 1.52, 1.25 (2H), 1.25, 0.55 (s, CH₃), 0.80 (s, CH₃), 2.05, 1.03 (d, CH₃, J=6.8 Hz), 5.16 (dd, J=8.8 Hz & 15.2 Hz), 5.02 (dd, J=8.4 Hz & 15.2 Hz), 1.55, 1.55, 0.85 (CH₃, J= 6.4 Hz), 0.84 (d, CH₃, J= 6.0 Hz), 1.18, 1.42, 0.82 (t, CH₃ J=7.2 Hz).13CMR (δ CDCl₃, 100 MHz): 37.2 (C-1), 31.5 (C-2), 71.1 (C-3), 38.0 (C-4), 40.3 (C-5), 29.7 (C-6), 117.5 (C-7), 139.6 (C-8), 49.5 (C-9), 34.2 (C-10), 21.6 (C-11), 39.6 (C-12), 43.3 (C-13), 55.1 (C-14), 23.0 (C-15), 28.5 (C-16), 55.9 (C-17), 12.0 (C-18), 13.0 (C-19), 40.8 (C-20), 21.4 (C-21), 138.1 (C-22), 129.5 (C-23), 51.2 (C-24), 31.9 (C-25), 21.1 (C-26), 19.0 (C-27), 25.4 (C-28), 12.2 (C-29).MS: 412.69 [M⁺]**Spinasteryl- β -D-glucopyranoside** (Boonsri, 2004; Rashid *et al.*, 2012)Molecular Formula: C₃₅H₅₈O₆UV (λ_{max} , MeOH, nm): 205.2.IR (KBr) cm⁻¹: 1091, 1374, 1472, 2897, 3403.¹HNMR (δ Pyridine-d5+CDCl₃): 1.70 (1H, m, H-1), 0.94 (1H, m, H-1), 1.94 (1H, m, H_a), 1.32 (1H, m, H_b), 3.93 (1H, m, H-3), 2.00 (1H, m, H_a), 1.55 (1H, m, H_b), 1.22 (1H, m, H-5), 1.69 (2H, m, H-6), 5.17 (1H, br. m, H-7), 1.60 (1H, m, H-9), 1.55 (1H, m, H-11), 1.43 (1H, m, H-11), 1.97 (2H, m, H-12), 1.82 (1H, m, H-14), 1.79 (1H, m, H-15), 1.56 (1H, m, H-16), 1.30 (2H, m, H-16), 1.28 (1H, m, H-17), 0.58 (3H, s, H-18), 0.72 (3H, s, H-19), 2.06 (1H, m, H-20), 1.08 (3H, d, J=6.5 Hz, H-21), 5.21(1H, dd, J=15.5 Hz & 9.0 Hz, H-22), 5.07 (1H, dd, J=15.5 Hz & 9.0 Hz, H-23), 1.57 (1H, m, H-24), 1.56 (1H, m, H-25), 0.86 (3H, d, J=7.0 Hz, H-26), 0.91 (3H, d, J=6.5 Hz, H-27), 1.42 (2H, m, H-28), 0.88 (3H, t, J=7.5 Hz, H-29), 4.93 (1H, d, J=8.0 Hz, H-1'), 3.91 (1H, m, H-2'), 4.18 (1H, t, J=8.5 Hz, H-3'), 4.13 (1H, t, J=8.5 Hz, H-4'), 3.89 (1H, m, H-5'), 4.48 (1H, br. d, J=12.0 Hz, H_a-6'), 4.31 (1H, brdd, J=12.00 Hz & 5.5 Hz, H_b-6'').13CMR (δ Pyridine-d5+CDCl₃): 35.13 (C-1), 32.40 (C-2), 75.03 (C-3), 27.69 (C-4), 37.97 (C-5), 27.77 (C-6), 115.62 (C-7), 137.29 (C-8), 47.37 (C-9), 32.33 (C-10), 19.52 (C-11), 37.44 (C-12), 41.26 (C-13), 53.10 (C-14), 21.12 (C-15), 26.68 (C-16), 53.84 (C-17), 10.09 (C-18), 10.91 (C-19), 38.94 (C-20), 19.46 (C-21), 136.42 (C-22), 137.42 (C-23), 49.27 (C-24), 29.96 (C-25), 17.05 (C-26), 19.17 (C-27), 23.50 (C-28), 10.36(C-29), 99.91 (C-1'), 72.80 (C-2'), 76.05 (C-3'), 69.43 (C-4'), 75.90 (C-5'), 60.61 (C-6').MS: 574.84 [M⁺] **α -Spinasterol** (Billah *et al.*, 2013)Molecular Formula: C₂₉H₄₈OIR (KBr) cm⁻¹: 3420 (OH), 3050 (H-C=C), 2930, 3850, 1640, 1450, 1370, 1040, 970, 830.¹HNMR (δ CDCl₃): 0.540 (s, 3H in C-18), 0.795 (br. s, 3H in C-27), 0.795 (3H in C-29), 0.814 (s, 3H in C-19), 0.847 (d, J= 5.9 Hz in C-26), 1.024 (d, J= 6.67 Hz in C-21), 1.40 – 2.0 (m, for -CH & -CH₂ protons), δ 2.009 (s, oxygenated methine proton at C-3), 3.584 (m, 1H at C-3), 5.024 (m, 1H, H-7), 5.045 – 5.119 (m, 2H, H-22 and H-23).13CMR (CDCl₃, 100 MHz): 32.6 (C-1), 32.5 (C-2), 71.2 (C-3), 38.0 (C-4), 44.4 (C-5), 29.4 (C-6), 121.9 (C-7), 141.7 (C-8), 49.5 (C-9), 45.1 (C-10), 24.8 (C-11), 35.8 (C-12), 46.2 (C-13), 55.1 (C-14), 28.0 (C-15), 27.5 (C-16), 56.2 (C-17), 20.9 (C-18), 20.8 (C-19), 40.2 (C-20), 20.2 (C-21), 130.4 (C-22), 134.9 (C-23), 52.2 (C-24), 31.9 (C-25), 21.1 (C-26), 21.1 (C-27), 26.5 (C-28), 12.3 (C-29).MS: 412.69 [M⁺]**Stigmasterol** (Chaturvedula and Prakash, 2012)Molecular Formula: C₂₉H₄₈OUV (λ_{max} , nm): 257IR (CHCl₃) cm⁻¹: 3320, 2946, 2854, 1480, 1388, 1189, 1096, 1035, 668.¹HNMR (δ CDCl₃, 600MHz): 3.51 (tdd, 1H, J = 4.5, 4.2, 3.8 Hz), 5.31 (t, 1H, J = 6.1 Hz), 0.91 (d, 3H, J = 6.2 Hz), 4.98 (m, 1H), 5.14 (m, 1H), 0.83 (t, 3H, J = 7.1 Hz), 0.82 (d, 3H, J = 6.6 Hz), 0.80 (d, 3H, J = 6.6 Hz), 0.71 (s, 3H), 1.03 (s, 3H).13CMR (δ CDCl₃, 150MHz): 37.6 (C-1), 32.2 (C-2), 72.1 (C-3), 42.4 (C-4), 141.1 (C-5), 121.8 (C-6), 31.8 (C-7), 31.8 (C-8), 50.2 (C-9), 36.6 (C-10), 21.5 (C-11), 39.9 (C-12), 42.4 (C-13), 56.8 (C-14), 24.4 (C-15), 29.9 (C-16),

56.2 (C-17), 40.6 (C-18), 21.7 (C-19), 138.7 (C-20), 129.6 (C-21), 46.1 (C-22), 25.4 (C-23), 12.1 (C-24), 29.6 (C-25), 20.2 (C-26), 19.8 (C-27), 18.9 (C-28), 12.2 (C-29).

MS (*m/z*): 412 [M+], 394, 351, 314, 300, 271, 229, 213, 55

Bis (2-ethylhexyl) phthalate (Habib *et al.*, 2009; Amatya *et al.*, 2005)

Molecular Formula: C₂₄H₃₈O₄

UV (λ_{\max} , nm): 246.2, 273.4.

IR (KBr) cm⁻¹: 1739 (C=O), 1047-1250 (C-O).

¹HNMR (δ CDCl₃, 300 MHz): 0.91 (t, 6H, *J* = 6.6 Hz, 6-H, 2''-H); 1.20 – 1.50 (m, 2-H, 3-H, 4-H and 5-H, merged), 1.60 – 1.70 (q, 2H, 2'-H), 4.20 (dd like, 2H, 1-H), 7.51 (dd, 1H, *J* = 6.6, 3.3 Hz, 10-H), 7.68 (dd, 1H, *J* = 6.6 & 3.3 Hz, 9-H).

¹³CMR (δ CDCl₃, 75 MHz): 10.8 (C-6), 14.0 (C-2''), 23.6 (C-4), 22.9 (C-5), 28.8 (C-3), 30.2 (C-2''), 38.6 (C-2), 68.0 (C-1), 128.7 (C-10), 130.8 (C-9), 132.3 (C-8), 167.6 (C-7).

EI-MS (*m/z*, %): 390 [M]⁺ (0.8), 279 (28.8), 167 (43.2), 149 (100), 132 (2.4), 113 (11.2), 83 (6.4), 71 (18.4).

Serratumin A (Hui *et al.*, 2000)

Molecular Formula: C₁₆H₂₂O₈

UV (λ_{\max} , MeOH nm): 202, 214.5, 253.5, 258.5.

IR (KBr) cm⁻¹: 3418 (broad), 3020, 2936, 1704, 1642, 1488, 1425, 1385, 1248, 1086, 755, 682. ¹HNMR (δ Pyridine-d₅, 500MHz): 6.93 (br. d, *J* = 5.8 Hz, H-3), 2.19 (2H, m, H-4), 2.19 (2H, m, H-5), 3.86 (1H, t, *J*=9.2 Hz, H-7 β), 4.54 (2H, m, H-8), 5.02 (1H, br. s, H-9a), 5.14 (1H, br. s, H-9b), 1.91 (3H, s, H-10), 5.55 (1H, s, H-1a), 5.37 (1H, d, *J*=10.0 Hz, H-4' β), 3.92 (1H, dt, *J*=10.0 Hz & 1.8 Hz), 4.46 (2H, br. s H-6').

¹³CMR (δ Pyridine-d₅, 100MHz): 170.5 (s, C-1), 129.8 (s, C-2), 140.9 (d, C-3), 27.40 (t, C-4), 35.44 (t, C-5), 141.9 (s, C-6), 51.80 (d, C-7 β), 71.96 (t, C-8), 114.9 (t, C-9), 12.95 (q, C-10), 110.4 (d, C-1'a), 87.38 (s, C-2''), 209.6 (s, C-3''), 72.58 (d, C-4 β), 78.74 (d, C-5 α), 62.27 (t, C-6').

EI-MS (*m/z* %): 324 [M-H₂O]⁺ (20), 306 [324 -H₂O]⁺ (24), 294 (15), 264 (16), 235 (17), 217 (20), 206 (30), 193 (22), 177 (54), 166 (62), 148 (62), 121 (160), 107 (58).

Serratoside A (Yang *et al.*, 2000b)

Molecular Formula: C₂₅H₂₈O₁₁

UV (λ_{\max} MeOH (log ϵ) nm): 203.5 (5.15), 216.5 (5.13), 22.5 (5.14), 243.5 (5.13), 256.5 (5.09), 278 (5.20).

IR (CHCl₃) cm⁻¹: 3391(br), 1712, 1675, 1624, 1450, 1355, 1276, 1171, 869. ¹HNMR (δ pyridine-d₅, 400MHz): 6.38 (H-1a, d, *J*=2.3Hz), 7.48 (H-3, s), 3.56 (H-6a, dd, *J*=13.2 Hz & 7.6 Hz), 2.60 (H-6 β , dd, *J*=13.0 Hz & 8.3 Hz), 5.76 (br H-7a, t, *J*=5.8 Hz), 3.62 (br H-9 β , d, *J*=2.3 Hz), 5.28 (H-10a br, s), 5.49 (H-10b br, s), 9.45 (H-11, s), 5.37 (H-1',d, *J*=7.8 Hz), 4.05 (H-2', t, *J*=8.3 Hz), 4.23 (H-3',H-

4', m), 4.02 (H-5', m), 4.55 (H-6'a, dd, *J*=11.8 Hz & 2.0 Hz), 4.37 (H-6'b, dd, *J*=11.8 Hz & 5.5 Hz), 7.59 (H-2'',H-6'' br, d, *J*=7.5), 7.36 (H-3'', H-5''br, d, *J*=7.5 Hz), 7.35 (H-4'' br, s), 6.68 (H β , d, *J*=16.0 Hz), 7.88 (H γ , d, *J*=16.0 Hz)

¹³CMR (δ CDCl₃): 97.27 (C-1,d), 162.77 (C-3, d), 123.98 (C-4, s), 70.39 (C-5, s), 42.91 (C-6, t), 73.70 (C-7, d), 146.57 (C-8, s), 52.94 (C-9, d), 114.38 (C-10, t), 190.58 (C-11, s), 100.82 (C-1', d), 74.63 (C-2', d), 79.13 (C-3', d), 71.52 (C-4', d), 78.39 (C-5', d), 62.70 (C-6', t), 134.95 (C-1'', s), 128.68 (C-2'' and C-6'', d), 129.40 (C-3'' and C-5'', d), 130.81 (C-4'', d), 166.76 (C_a, s), 118.89 (C_b, D), 145.33s (C_y, d).

MS (FABMAS): [M+1]⁺505.1714.

Serratoside B (Yang *et al.*, 2000b)

Molecular Formula: C₂₅H₂₈O₁₁

UV (λ_{\max} MeOH (log ϵ) nm,): 203.5 (5.15), 216.5 (5.13), 22.5 (5.14), 243.5 (5.13), 256.5 (5.09), 278 (5.20).

IR (CHCl₃), cm⁻¹: 3391(br), 1712, 1675, 1624, 1450, 1355, 1276, 1171, 869.

¹HNMR (δ Pyridine-d₅, 400MHz): 5.83 (H-1 β , s, *J*=7.5 Hz & 7.50 Hz), 7.50 (H-3, s), 2.88 (H-6a, br. d, *J*=17.3 Hz), 3.27 (H-6 β , br. d, *J*=17.3 Hz), 5.78 (H-7, br. s), 3.47 (H-9 β , d, *J*=7.0 Hz), 5.11 (H-10a, d, *J*=13.9 Hz), 5.22 (H-10b, d, *J*=13.9 Hz), 9.50 (H-11, s), 5.43 (H-1',d, *J*=7.8 Hz), 4.14 (H-2', t, *J*=8.0 Hz), 4.28 (H-3', H-4', m), 4.03 (H-5', m), 4.54 (H-6'a, dd, *J*=11.8 Hz & 2.0 Hz), 4.35 (H-6'b, dd, *J*=11.8 Hz & 5.7 Hz), 7.55 (H-2'',H-6'', d, *J*=6.4 Hz), 7.34 (H-3'', H-5'', d, *J*=6.4 Hz), 7.33 (H-4'', s), 6.67 (H β , d, *J*=16.0 Hz), 7.86 (H γ , d, *J*=16.0 Hz)

¹³CMR (δ CDCl₃): 99.64 (C-1,d), 161.46 (C-3, d), 126.59 (C-4, s), 75.34 (C-5, s), 44.76 (C-6, t), 129.30 (C-7, d), 136.71 (C-8, s), 57.21 (C-9, d), 62.82 (C-10, t), 190.57 (C-11, s), 101.21 (C-1', d), 74.80 (C-2', d), 78.99 (C-3', d), 71.52 (C-4', d), 78.41 (C-5', d), 62.82 (C-6', t), 134.96 (C-1'', s), 128.66 (C-2'' and C-6'', d), 129.30 (C-3'' and C-5'', d), 130.68 (C-4'', d), 166.63 (C_a, s), 118.76 (C_b, D), 145.19 (C_y, d).

MS (FABMAS): [M+1]⁺505.1666.

(+)-Catechin (Hye *et al.*, 2009)

Molecular Formula: C₁₅H₁₄O₆

UV (λ_{\max} MeOH nm): 277.220.

IR (KBr) cm⁻¹: 2600-3400 (broad), 1620, 1520, 1470, 1380, 1280, 1240, 1150, 1120, 1080, 1020, 820.

¹HNMR (δ Acetone-d₆, 400 MHz): 4.56 [H-2, d, *J*_(H-2, H-3a) 7.8 Hz], 4.00 [H-3, ddd, *J*_(H-3a, H-4e) 5.58 Hz, *J*_(H-3a, H-4a) 8.50 Hz, *J*_(H-3a, H-2a) 7.80 Hz], 2.54 [H-4a, dd, *J*_(H-4a, H-3a) 8.50 Hz, *J*_(H-4a, H-4e) 16.10 Hz], 2.90 [H-4e, dd, *J*_(H-4e, H-3a) 5.50 Hz, *J*_(H-4e, H-4a) 16.10 Hz], 5.87 [H-6, d, *J*_(H-6, H-8) 2.3 Hz], 6.01 [H-8, d, *J*_(H-8, H-6) 2.3 Hz], 6.89 [H-2', d, *J*_(H-2', H-6') 1.95 Hz], 6.79 [H-5', d, *J*_(H-5', H-6') 8.07 Hz], 6.73 [H-6', dd, *J*_(H-6', H-2) 1.94 Hz, *J*_(H-6', H-5') 8.19 Hz] and 8.00 (phenolic protons, m).

¹³CMR (δ DMSO, 500 MHz): 27.7 (C-4), 66.3 (C-3), 80.9 (C-2), 93.9 (C-6), 95.1 (C-8), 114.5 (C-2 \circlearrowleft), 115.1 (C-5 \circlearrowright), 18.4 (C-6 \circlearrowright), aromatic carbons show peaks at δ of 99.1, 130.6, 144.6, 144.8, 155.3, 156.1 and 156.4.
MS (m/z): 290 [M⁺], 139, 138, 110, 152, 151, 123, 55.

Caffeic acid (Bhatt, 2011)

Molecular Formula: C₉H₈O₄

UV (λ_{max} , MeOH nm): 240 (4.18), 280 (4.17) and 350, sh (3.60).

IR (KBr) cm⁻¹: 3368.42, 2650.20, 1680.87, 1610.25, 1593.15, 1510.30, 1123.18, 760.98, 715.34.

¹HNMR (δ D₂O, 300 MHz): 7.511 (1H, d, J = 15.0 Hz, H-7), 7.101 (1H, s, H-2), 7.029 (1H, d, J = 8.0 Hz, H-6), 6.807 (1H, d, J = 8.0 Hz, H-5), 6.351 (1H, d, J = 15.0 Hz, H-8).

¹³CMR (δ CDCl₃, 75 MHz): 125.42 (C-1), 114.86 (C-2), 145.21 (C-3), 148.35 (C-4), 115.75 (C-5), 121.35 (C-6), 141.41 (C-7), 127.50 (C-8), 174.65 (C-9).

MS (m/z): 180.08[M⁺], 163.06, 135.08, 109.08, 92.06, 80.09, 75.07, 65.07.

Ferulic acid (Sajjadi et al., 2012)

Molecular Formula: C₁₀H₁₀O₄

UV (λ_{max} , nm): 321.

IR (KBr) cm⁻¹: 3450, 1690, 1605, 1510, 1275, 940.

¹HNMR (δ CDCl₃, 500 MHz): 3.98 (3H, s, H-4'), 6.34 (1H, d, J =15 Hz, H-2'), 6.97 (1H, d, J =9 Hz, H-6), 7.14 (1H, dd, J =8 Hz & 2 Hz, H-5), 7.09 (1H, d, J =2 Hz, H-3), 7.75 (1H, d, J =15 Hz, H-1').

¹³CMR (δ CDCl₃, 500 MHz): 55.98 (C-4'), 109.48 (C-5), 114.39 (C-2), 114.78 (C-2'), 123.57 (C-3), 126.68 (C-4), 146.81 (C-1'), 147.05 (C-6), 148.37 (C-1), 171.36 (C-3').

EIMS (m/z, relative intensity): 194 [M]⁺ (100), 179 (21), 161 (7), 133 (32), 105 (14), 89 (15), 77 (27), 51 (15).

Serratoside-A (Yang et al., 2000b)

Molecular Formula: C₃₆H₄₈O₁₉

UV (λ_{max} (log ϵ) nm): 214.5 (5.15), 258 (4.73), 282.0 (4.59), 325.5. (5.21).

IR (KBr) cm⁻¹: 3417 (br), 1705, 1630, 1595.

¹HNMR (δ Pyridine-d₅, 500 MHz): Aglycone proton: 6.83 (H-2, d, J =1.8 Hz), 6.71 (H-5, d, J =8.1 Hz), 6.67 (H-6, dd, J =8.1 Hz & J =1.9 Hz), 3.89 (Haa, m), 3.67 (Hab, m), 2.76 (H- β , m), 3.75 (H-OMe, s). Acyl moiety: 7.32 (H-2, d, J =1.8 Hz), 6.84 (H-5, d, J =8.1 Hz), 7.12 (H-6, dd, J =8.0 Hz, J =1.8 Hz), 6.44 (H- β' , d, J =15.8 Hz), 7.58 (H- γ' , d, J =15.8 Hz), 3.83 (H-OMe, s). Glucosyl moiety: 4.41 (H-1, d, J =7.6 Hz), 3.20 (H-2, m), 3.72 (H-3, H-5, m), 4.71 (H-4, t, J =9.7 Hz), 3.45 (H-6a, m), 3.40 (H-6b, m).

Rhamnosyl moiety: 5.06 (H-1, br. s), 3.72 (H-2, H-3, m), 3.14 (H-4, m), 3.37 (H-5, m), 1.01 (H-6, d, J =6.1 Hz).

Apiosyl moiety: 4.81 (H-1, d, J =2.7 Hz), 3.72 (H-2, m), 3.80 (H-4a, d, J =9.2 Hz), 3.58 (H-4b, d, J =9.2 Hz), 3.32 (H-5, m).

¹³CMR (δ CDCl₃): Aglycone moiety: 131.1 (C-1, s), 112.4 (C-2, d), 146.2 (C-3, s), 145.9 (C-4, s), 116.4 (C-5, d), 119.6 (C-6, d), 70.2 (C- α , t), 35.0 (C- β , t), 55.8 (C-OMe, q). Acyl moiety: 125.8 (C-1, s), 111.2 (C-2, d), 148.0 (C-3, s), 149.5 (C-4, s), 115.6 (C-5, d), 123.3 (C-6, d), 165.9 (C- α' , s), 114.0 (C- β' , d), 146.3 (C- γ' , d), 55.8 (C-OMe, q). Glucosyl moiety: 102.3 (C-1, d), 74.5 (C-2, d), 78.9 (C-3, d), 69.5 (C-4, d), 72.9 (C-5, d), 67.2 (C-6, t). Rhamnosyl moiety: 101.3 (C-1, d), 70.6 (C-2, d), 76.1 (C-3, d), 71.7 (C-4, d), 68.9 (C-5, d), 18.2 (C-6, q). Apiosyl moiety: 109.2 (C-1, d), 76.1 (C-2, d), 78.9 (C-3, s), 73.5 (C-4, t), 63.2 (C-5, t). MS (FAB-MS): 783.2640 [M-1]⁻

Acetoside (Chuan-Ling et al., 2011; Tatlı et al., 2007; Ersoz et al., 2002a)

Molecular Formula: C₂₉H₃₆O₁₅

UV (λ_{max} , MeOH, nm): 212, 332.

IR (KBr) cm⁻¹: 3689 (OH), 1708 (C=O), 1634 (C=C), 1604, 1515, 1385 (aromatic ring).

¹HNMR (δ CD₃OD, 500 MHz): Aglycone -6.69 (d, J =1.8 Hz, H-2), 6.67 (d, J =8.2 Hz, H-5), 6.56 (dd, J =8.2 Hz & 1.8 Hz, H-6), 4.05 (m, H- α), 3.72 (m, H- α), 2.79 (t, J =7.2 Hz, H- β), Glucose moiety- 4.37 (d, J =7.9 Hz, H-1'), 3.39 (dd, J =9.1 Hz & 7.2 Hz, H-2'), 3.81 (t, J =9.1 Hz, H-3'), 4.95 (t, J =9.4 Hz, H-4'), 3.55 (m, H-5'), 3.61 (dd, J =12.2 Hz & 2.0 Hz, H-6'), 3.53 (dd, J =12.2 Hz & 6.4Hz, H-6'), Rhamnose moiety- 5.18 (d, J =1.8 Hz, H-1''), 3.91 (dd, J =3.4 Hz & 1.8 Hz, H-2''), 3.57 (dd, J =9.7 Hz & 3.4 Hz, H-3''), 3.28 (t, J =9.7 Hz, H-5''), 1.09 (d, J =6.2 Hz, H-6''), Aceyl moiety- 7.05 (d, J =1.04 Hz, H-2'''), 6.77 (d, J =8.2 Hz, H-5'''), 6.96 (dd, J =8.2 Hz & 1.4 Hz, H-6'''), 6.28 (d, J =15.9 Hz, H- α '), 7.59 (d, J =15.9 Hz, H- β ').

¹³CMR (δ CD₃OD, 125 MHz): 131.5 (C-1), 117.2 (C-2), 146.7 (C-3), 144.3 (C-4), 116.4 (C-5), 121.3 (C-6), 72.4 (C- α), 36.6 (C- β), Glucose moiety- 104.3 (C-1'), 76.3 (C-2'), 81.7 (C-3'), 70.7 (C-4'), 76.1 (C-5'), 62.4 (C-6'), Rhamnose moiety- 103.1 (C-1''), 72.3 (C-2''), 72.1 (C-3''), 73.9 (C-4''), 70.5 (C-5''), 18.5 (C-6''), Aceyl moiety- 127.7 (C-1'''), 115.3 (C-2'''), 146.9 (C-3'''), 149.9 (C-4'''), 116.6 (C-5'''), 123.2 (C-6'''), 114.8 (C- α '), 148.1 (C- β '), 168.3 (C=O). MS (m/z): [M+H]⁺ 625 and [M+Na]⁺ 647.

Martynoside (Ersoz et al., 2002a)

Molecular Formula: C₃₁H₄₀O₁₅

UV (λ_{max} , MeOH nm): 330, 287, 220.

IR (KBr) cm⁻¹: 3400 (OH), 1700 (α , β -unsaturated ester), 1625 (olefinic C=C), 1605, 1515 (arom. ring).

¹HNMR (δ CD₃OD, 300.13 MHz): Aglycone -6.74 (d, J =2.1 Hz, H-2), 6.82 (d, J =8.2 Hz, H-5), 6.69 (dd, J =8.2 Hz & 2.1 Hz, H-6), 4.05 (m, H- α), 3.75 (m, H- α), 2.83 (m, H- β), 3.82 (s, OMe), Glucose moiety- 4.38 (d, J =7.9 Hz, H-1'), 3.29 (dd, J =7.9 Hz & 9.5 Hz, H-2'), 3.85 (t,

$J=9.5$ Hz, H-3'), 4.95 (t, $J=9.5$ Hz, H-4'), 3.53 (H-5'), 3.66 (dd, $J=11.9$ Hz & 6.4 Hz, H-6'), 3.52 (dd, $J=11.9$ Hz & 2.3 Hz, H-6'), Rhamnose moiety- 5.20 (d, $J=1.5$ Hz, H-1''), 3.92 (dd, $J=3.2$ Hz & 1.7Hz, H-2''), 3.63 (t, $J=9.9$ Hz, H-3''), 3.29 (t, $J=9.5$ Hz, H-4''), 3.55 (m, H-5''), 1.10 (d, $J=6.2$ Hz, H-6''), Aceyl moiety- 7.20 (d, $J=1.8$ Hz, H-2''), 6.83 (d, $J=8.2$ Hz, H-5''), 7.09 (dd, $J=8.2$ Hz & 1.8 Hz, H-6''), 6.39 (d, $J=15.9$ Hz, H- α'), 7.67 (d, $J=15.9$ Hz, H- β'), 3.89 (s, C=O).

^{13}CMR (δ CD₃OD, 75.5 MHz): 132.9 (C-1), 112.8 (C-2), 147.9 (C-3), 147.6 (C-4), 117.1 (C-5), 121.2 (C-6), 72.4 (C- α), 36.6 (C- β), 56.5 (OMe), Glucose moiety-104.2 (C-1'), 76.2 (C-2'), 81.5 (C-3'), 70.6 (C-4'), 76.1 (C-5'), 62.4 (C-6'), Rhamnose moiety- 103.0 (C-1''), 72.1 (C-2''), 72.0 (C-3''), 73.8 (C-4''), 70.4 (C-5''), 18.4 (C-6''), Aceyl moiety- 127.6 (C-1''), 111.7 (C-2''), 149.4 (C-3''), 150.8 (C-4''), 116.5(C-5''), 124.4 (C-6''), 115.1 (C- α'), 147.9 (C- β'), 168.3 (C=O), 56.4 (OMe). MS: 652.23 [M]+

Apigenin (Owen *et al.*, 2003; Ersöz *et al.*, 2002b; Modnicki *et al.*, 2007)

Molecular Formula: C₁₅H₁₀O₅
UV (λ_{max} , nm): 265, 297, 335.

IR (KBr) cm⁻¹: 3297-3095 (OH), 2922-2617 (CH), 1654 (C=O), 1608, 1500 (aromatic rings), 1445, 1354, 1298, 1267.

$^1\text{HNMR}$ (δ CD₃OD, 500 MHz): 7.83 (2H, d, $J = 8.8$ Hz, H-2' and H-6'), 6.92 (2H, d, $J = 8.8$ Hz, H-3' and H-5'), 6.83 (1H, d, $J = 2.1$ Hz, H-6), 6.71 (1H, d, $J = 2.1$ Hz, H-8), 6.58 (1H, s, H-3).

^{13}CMR (δ CD₃OD, 125 MHz): 180.45 (s, C-4), 164.94 (s, C-5), 164.41 (s, C-2), 162.60 (s, C-40), 160.72 (s, C-9), 160.16 (s, C-7), 129.30 (d, C-2' and C-6'), 123.14 (s, C-1'), 117.06 (d, C-3' and C-5'), 109.39 (s, C-1'), 106.57 (d, C-3), 104.83 (d, C-6), 99.34 (d, C-8).

MS (m/z & Intensity): 485 [M]⁺, 471(100), 399(5), 228(9).

Apigenin-7-glucoside (Bhujbal *et al.*, 2010b)

Molecular Formula: C₂₁H₂₀O₁₀
UV (λ_{max} , EtOH nm): 247, 352.

IR (KBr) cm⁻¹: 3402, 2920, 2850, 1631, 597.

$^1\text{HNMR}$ (δ DMSO-d6, 300 MHz): 3.5 (t, $J=3.6$, H-3, CH), 3.8 (t, $J=1.8$, 2-H, CH₂OH), 4.0 (q, $J=31.5$, 1-H, CH-O), 4.7 (t, $J=19.5$, 1-H, CH₂OH), 4.9 (d, $J=22.5$, 3-H, OH), 6.0 (d, $J=18.3$, 1-H, CH-O), 6.7 (s, 3-H, Ar C-H), 7.1 (d, $J=6.9$ 4-H, Ar C-H), 8.5 (s, 2-H, Ar-OH).

^{13}CMR (δ CDCl₃): 79.0(C-2), 43.1 (C-3), 196.9 (C-4), 161.1 (C-5), 105.3 (C-6), 145.1 (C-7), 104.8 (C-8), 37.4 (C-9), 133.3 (C-1'), 128.6 (C-2' & C-6'), 116.1 (C-3' & C-5'), 157.4 (C-4'), 74.0 (C-1''), 75.1 (C-2''), 77.1 (C-3''), 71.5 (C-4''), 81.4 (C-5''), 62.3 (C-6'').

MS (FAB-MS): 433 [M+1]⁺, 432 [M]⁺, 271 (glucose residue)

Luteolin (Chaturvedula *et al.*, 2013; Đorđević *et al.*, 2000)

Molecular Formula: C₁₅H₁₀O₆
UV (λ_{max} , MeOH nm): 253, 268, (290), 348; + NaOMe: 265, (330), 402; + AlCl₃: 274, (300), (329), 425; +AlCl₃/HCl: 275, 296, 356, 388; + NaOAc: 271, (325), 390; +NaOAc/H₃BO₃: 262, (301), 372, (430). IR (KBr) cm⁻¹: 3490-3400 (OH), 2923-2617 (C-H), 1655 (C=O), 1610-1490 (aromatic rings), 1458, 1364, 1267.

$^1\text{HNMR}$ (δ C₅D₅N, 600MHz): 7.56 (1H, dd, $J = 9$, 2Hz, H-6'), 7.36 (1H, d, $J = 2$ Hz, H-2'), 6.85 (1H, d, $J = 9$ Hz, H-5'), 6.75 (1H, s, H- 3), 6.46 (1H, d, $J = 2$ Hz, H-8) , 6.28 (1H, d, $J = 2$ Hz, H-6).

^{13}CMR (δ C₅D₅N, 125 Hz): 181.8 (C-4), 164.3 (C-7), 164.0 (C-2), 162.1 (C-9), 157.6 (C-5), 149.7 (C- 4'), 146.0 (C-3'), 120.8 (C-6'), 119.0 (C-1'), 116.8 (C-5'), 113.2 (C-2'), 103.8 (C-10), 99.2 (C-6), 94.7 (C-8)

ESI (m/z): [M+H]⁺ 287.

Luteoline 7-O- β -D-glucuronide (Iwashina *et al.*, 2011; Modnicki *et al.*, 2007)

Molecular Formula: C₂₁H₁₈O₁₂
UV (λ_{max} , MeOH nm): 255, 265sh, 349; +NaOMe 266, 390 (inc.); +AlCl₃ 274, 426; +AlCl₃/HCl 264sh, 273, 294sh, 360,385; +NaOAc 260, 403; +NaOAc/H₃BO₃ 260, 372.

IR (KBr) cm⁻¹: 3421 (OH), 2956-2854 (C-H), 1736 (COOH), 1650 (C=O), 1603-1460 (aromatic rings), 1379, 1261, 1174, 1122, 1075.

$^1\text{HNMR}$ (δ pyridine-d5,600 MHz): d 7.82 (1H, d, $J=2.3$ Hz, H-2'), 7.51 (1H, dd, $J=1.5$ and 8.3 Hz, H-6'), 7.26 (1H, d, $J=8.3$ Hz, H-5'), 7.04 (1H, d, $J=1.7$ Hz, H-8), 6.82(1H, s, H-3), 6.77 (1H, d, $J=2.1$ Hz, H-6), 5.73 (1H, d, $J=7.2$ Hz, glucuronyl H-1), 4.54 (1H, t, $J=8.4$ Hz, glucuronyl H-5), 4.32 (2H, t, $J=8.8$ Hz, glucuronyl H-3, H-4), 4.24 (1H, m, glucuronyl H-2).

^{13}CMR (δ pyridine-d5, 150 MHz): (luteolin) 165.6 (C-2), 104.0 (C-3), 183.1 (C-4), 162.1 (C-5), 100.9 (C-6), 164.2 (C-7), 95.6 (C-8), 158.1 (C-9), 104.0 (C-10), 122.8 (C-1'),114.5 (C-2'), 147.5 (C-3'), 151.6 (C-4'), 116.9(C-5'), 120.0 (C-6'); (glucuronic acid) 101.8(C-1), 74.5 (C-2), 77.8 (C-3), 73.5 (C-4), 76.5(C-5), 174.7 (C-6). MS (m/z): 463 [M+H]⁺

Scutellarein (Scotti *et al.*, 2011; Qian *et al.*, 2012; Verma *et al.*, 2012)

Molecular Formula: C₂₁H₁₈O₁₂
UV (λ_{max} , EtOH, nm): 286, 339 (e 16600; 18300)
IR (KBr) cm⁻¹: 3400 (-OH), 1710 (C=O).

$^1\text{HNMR}$ (δ DMSO-d6, 300 MHz): 6.78 (1H, s), 6.73 (1H, s), 6.90–6.93 (2H, d, $J = 8.8$ Hz), 7.90–7.93 (2H, d, $J = 8.8$ Hz), 8.71 (1H, s), 10.30 (1H, s), 10.44 (1H, s), 12.79 (1H, s).

^{13}CMR (δ):164.2 (C-2), 102.9 (C-3), 182.7 (C-4), 154.0 (C-5), 129.9 (C-6), 147.7 (C-7), 94.6 (C-8), 150.4 (C-9),

104.7 (C10), 122.2 (C-1'), 129.1 (C-2'), 116.6 (C-3'), 161.7 (C-4'), 116.6 (C-5'), 129.1 (C-6').
ESI-MS (m/z): 285 [M-H]⁻

Baicalein (Huang *et al.*, 2003)

Molecular Formula: C₁₅H₁₀O₅

UV (λ_{max} , EtOH (log ε) nm): 326 (4.17), 276 (4.42), 215 (4.49).

IR (KBr) cm⁻¹: 3411, 1654.

¹HNMR (δ DMSO-d6): 6.61 (1H, s), 6.92 (1H, s), 7.56 (3H, m), 8.05 (2H, d, J=58.1 Hz), 8.81 (1H, s), 10.57 (1H, s), 12.65 (1H, s).

¹³CMR (δ): 163.7 (C-2), 104.5 (C-3), 182.2 (C-4), 154.5 (C-5), 130.7(C-6), 155.2 (C-7), 99.4 (C-8), 130.4 (C-1'), 126.4 (C-2' & C-6'), 128.7 (C-3' C-5'), 128.0 (C-4').

MS: 270 [M]⁺

5-hydroxy-7,4-dimethoxy flavone (Kolak *et al.*, 2009)

Molecular Formula: C₁₇H₁₄O

UV (λ_{max} , MeOH nm): 268, 328; MeOH+ NaOMe: 288, 340; MeOH + AlCl₃: 277, 380; MeOH + AlCl₃ + HCl: 277, 380; MeOH + NaOAc: 268, 330; MeOH + NaOAc + H₃BO₃: 268, 332.

IR (KBr) cm⁻¹: 3404, 2629, 1653, 1618, 1301, 1250, 1165, 1031, 999, 947, 860.

¹HNMR (δ CDCl₃, 500 MHz): 12.80 (1H, br s, 5-OH), 7.85 (2H, dd, J = 1.95 & 6.83 Hz, H-2' and H-6'), 7.02 (2H, dd, J = 1.95 & 6.83 Hz, H-3' and H-5'), 6.58 (1H, s, H-3), 6.48 (1H, d, J = 2.44 Hz, H-8), 6.37 (1H, d, J = 2.44 Hz, H-6), 3.89 (3H, s, OMe), 3.88 (3H, s, OMe).

¹³CMR (δ CDCl₃, 125 MHz): 182.41 (s, C-4), 164.30 (s, C-2), 156.41 (s, C-9), 154.61 (s, C-4'), 154.02 (s, C-5), 148.14 (s, C-7), 124.03 (s, C-1'), 120.32 (d, C-2' and C-6'), 111.54 (d, C-3' and C-5'), 103.42 (s, C-10), 103.34 (d, C-3), 98.06 (d, C-6), 93.51 (d, C-8), 61.51 (q, OMe), 56.15 (q, OMe).

HRESI-MS (m/z): 298.0837.

D-mannitol (Garg and Verma, 1996)

Molecular Formula: C₆H₁₄O₆

UV (λ_{max} , nm): 262

IR (KBr) cm⁻¹: 1100, 2950, 3450-3550.

¹HNMR (δ D₂O, 400 MHz): 3.60 (2H, dd, J = 5.9 & 11.6 Hz, H-1, 6), 3.70 (2H, m, H-2, 5), 3.73 (2H, t, J = 8.8 Hz, H-3, 4), 3.81 (2H, br d, J = 11.6 Hz, H-1, 6).

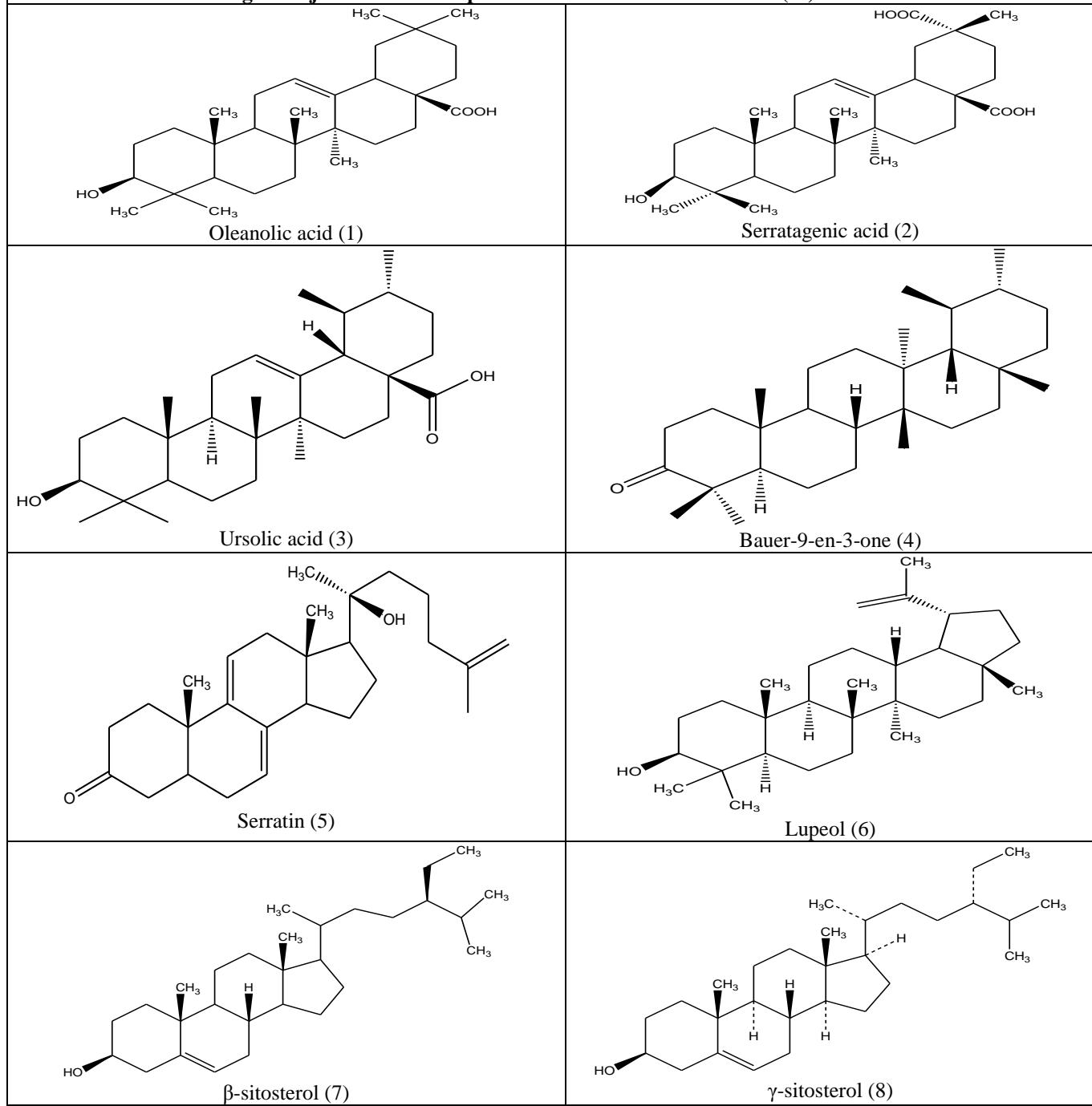
¹³CMR (δ, 100 MHz, CD₃OD): 65.98 (C-1, C-6), 72.01 (C-3, C-4), 73.57 (C-2, C-5).

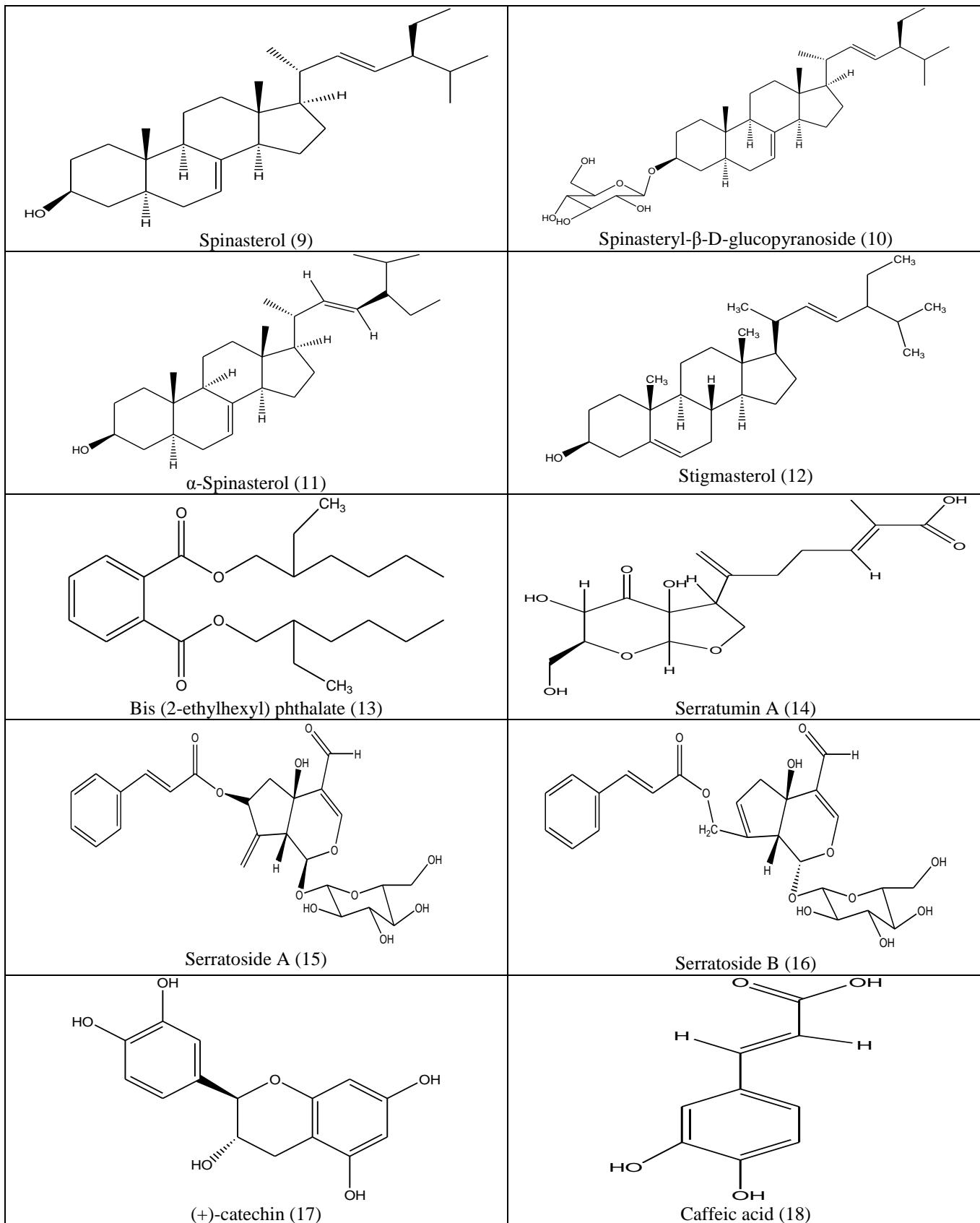
MS: m/z 183.2 [M]⁺

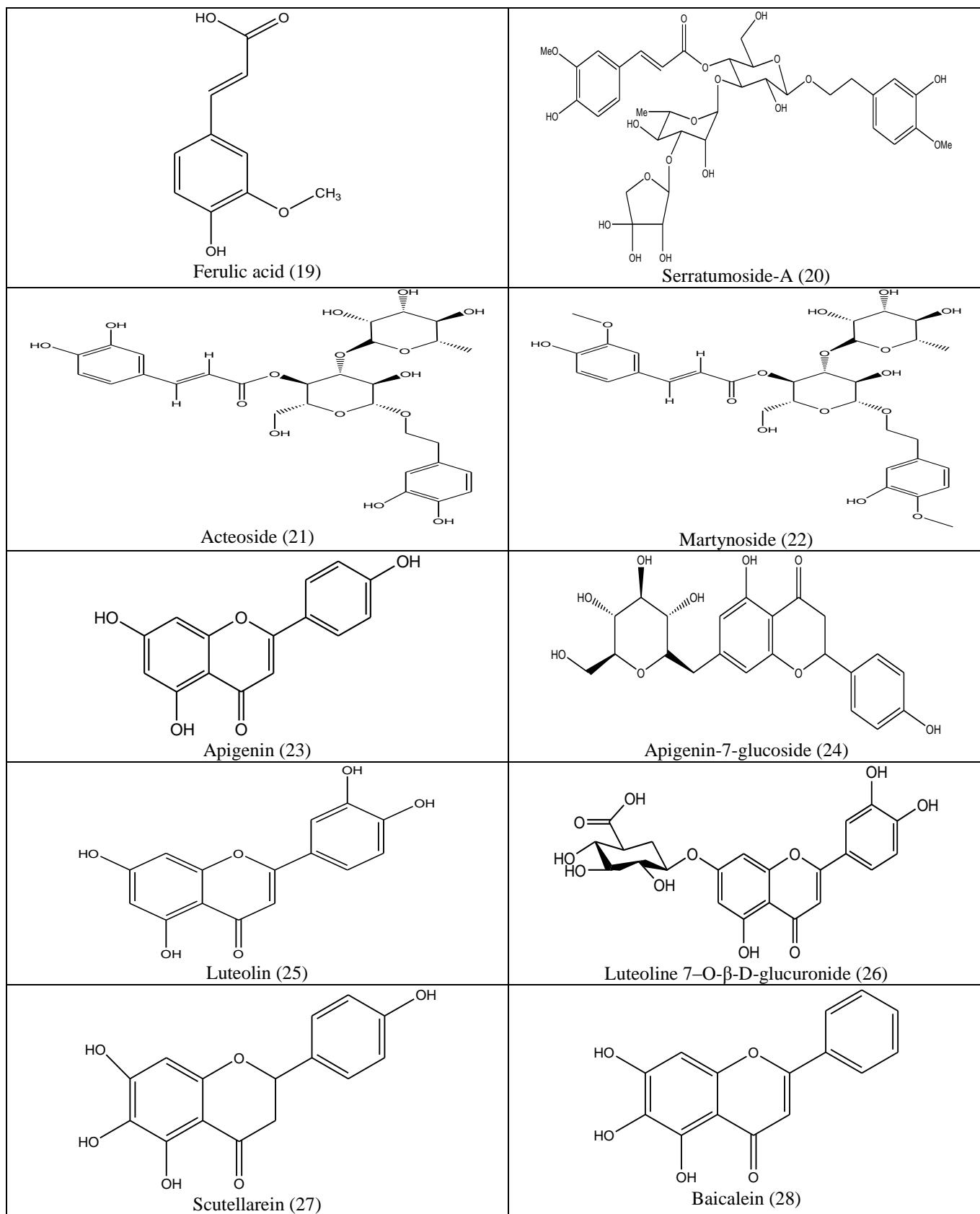
Table 1. Natural products isolated from *Clerodendrum serratum* (L.) Moon

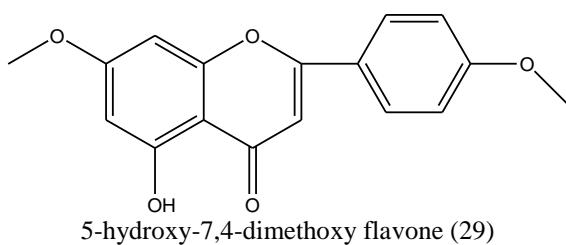
Chemical class	Chemical constituents	Plant part	References
Terpenoids	Oleanolic acid, Queretaroic acid and Serratogenic acid	Bark	Banerjee <i>et al.</i> , 1969; Rangaswami & Sarangan, 1969; Fan <i>et al.</i> , 2007
	Ursolic acid	Root and stem	Ganapaty <i>et al.</i> , 1997; Vidya <i>et al.</i> , 2007
	Icosahydropicenic acid	Root	Bhujbal <i>et al.</i> , 2010a
	Bauer-9-en-3-one	Twig and stem	Boonsri, 2004
	Se-saponin A	Aerial parts	Yang <i>et al.</i> , 2000b
	Serratin, Lupeol	Leaves	Raju <i>et al.</i> , 2008
Sterols	β -sitosterol	Stem	Ganapaty <i>et al.</i> , 1997
	γ -sitosterol	Root	Banerjee <i>et al.</i> , 1969
	Spinasterol, Spinasteryl- β -D-glucopyranoside	Twigs and stems	Boonsri, 2004
	α -spinasterol	Leaves	Nair <i>et al.</i> , 1976
	Stigmasterol, Bis(2-ethylhexyl) phthalate and Serratumin A	Aerial parts	Fan <i>et al.</i> , 2007
Iridoids	7- β -coumaroyl-oxyugandoside and 7- β -cinnamoyl-oxyugandoside	Leaves	Wei <i>et al.</i> , 2000
	Serratoside A and Serratoside B	Aerial parts	Yang <i>et al.</i> , 2000b
Phenyl propanoids	(+)-catechin, Caffeic acid and Ferulic acid	Leaves	Nair <i>et al.</i> , 1976
	Serratumoside-A and Myricoside	Aerial parts	Yang <i>et al.</i> , 2000a
	Acteoside and Martynoside	Aerial parts and Leaves	Yang <i>et al.</i> , 2000a; Fan <i>et al.</i> , 2007; Wei <i>et al.</i> , 2000
Flavonoids	Apigenin-7-glucoside	Root	Bhujbal <i>et al.</i> , 2010b
	Luteoline 7-0- β -D-glucuronide,	Leaves	Nair <i>et al.</i> , 1976

	Luteolin, Scutellarein, Apigenin, 6-hydroxyluteolin and Baicalein		
	5-hydroxy-7,4-dimethoxy flavones	Stem	Ganapaty <i>et al.</i> , 1997
	4',5,7-trihydroxy-flavone	Aerial parts	Fan <i>et al.</i> , 2007
Carbohydrate	Glucose and D-mannitol	Root	Garg & Verma, 1966
	Sucrose (disaccharide)	Twigs and stems	Boonsri, 2004

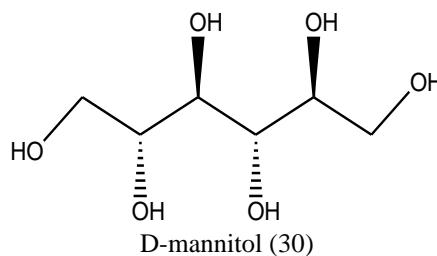
Fig 1. Major chemical compounds of *Clerodendrum serratum* (L.) Moon.







5-hydroxy-7,4-dimethoxy flavone (29)



D-mannitol (30)

CONCLUSION

Clerodendrum serratum is a widely distributed shrub in South-East Asia including India. More than 35 compounds consisting of different chemical classes have been identified till date. Present review discusses the phytochemistry and spectroscopic aspects. The plant is studied exhaustively in last 40 years. It is demonstrated the huge medicinal potential of *C. serratum*. The review describes analytical data for identified chemical compounds including different classes like terpenoids, sterols, iridoids, phenyl propanoids, flavonoids and carbohydrates. The spectroscopic details viz. Ultra-violet, Infrared, Mass and Nuclear magnetic resonance spectroscopic data have been compiled and represented.

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Nature is a unique source of structures of high phytochemical diversity, many of them possessing interesting biological activities and medicinal properties. This plant has its folklore uses in different traditional systems. Current review is extensively beneficial for modern ethnomedical practitioners to assess its potency scientifically with relevance to phytochemistry. The review helps to many phytochemical scientist for bioassay guided fractionation and isolation of many compounds.

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