Preparation, Characterization and Antimicrobial and Antifungal activities of 2- Methyl-4-(7-methoxy-2-oxo-2H-chromen-8-yl)-but-2-en-1-al, an Analogue of Osthol, a Major Constituent from Prangos pabularia

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ABSTRACT
Osthol, 7-methoxy-8-(3-methylbut-2-enyl) coumarin, was isolated from the root parts of Prangos pabularia and was subjected to modification in the isopentenyl side chain to get an aldehyde 2- Methyl-4-(7-methoxy-2-oxo-2H-chromen-8-yl)-but-2-en-1-al. The structures of osthol and compound 1 were elucidated on the basis of MS, IR, 1H and 13C-NMR spectroscopy. Compound 1 on bio-evaluation displayed significant antimicrobial and antifungal activity.

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Results and Discussion

Osthol was obtained in the form of colourless needles by extensive chromatography of the petroleum ether extract of the roots of \textit{Prangos pabularia} over silica gel, using graded solvent systems. The compound, in its mass spectrum, showed molecular ion peak at \( m/e \) 244 C\(_{13}\)H\(_{24}\)O\(_{3}\). The compound gave violet colouration with alkaline hydroxylamine, followed by addition of ferric chloride, characteristic of coumarins [13]. UV-spectrum of the compound displayed absorption peaks at \( \lambda_{\text{max}} \) 257, 255, 247 nm. The IR spectrum showed prominent bands at 1681 cm\(^{-1}\) (carbonyl group). However, no twin peaks at 1386 & 1367 cm\(^{-1}\) observed in addition to the band at 1728 cm\(^{-1}\), due to lactone carbonyl group. The compound displayed the down field resonance signals at \( \delta \) 6.18(H, a, J=9.1 Hz, H-3), 7.38 and 7.10 due to aromatic protons \((1H, \, d, \, J=5.26(H, t, H-2)\). The \( ^{1}H \) NMR spectra of the compound displayed down field resonance signals due to olefinic protons at \( \delta \) 5.26(1H, t, H-2). The \( ^{13}C \) NMR spectrum, displayed down field resonance signals at \( \delta \) 21.8 (-CH\(_{2}\), C-3), 121.0 (-CH=, C-2'), 126.4 (C, C-4'), 152.3 (C, C-5), 112.4 (C, C-6), 159.9 (-C=O, C-7), 152.3 (C, C-8a). The rest of the other carbon signals showed good antimicrobial and antifungal activities in comparison with the parent molecule osthol.

Comparison of physical characteristics and spectral data of the compound, with that reported in literature [12], confirmed it to be Osthol.

Compound 1, 2-Methyl-4-(7-methoxy-2-oxo-2H-chromen-8-yl)-but-2-en-1-al, was obtained by the oxidation of osthol with selenium dioxide in acetic acid. In its IR spectrum, a prominent band at 1681 cm\(^{-1}\), due to unsaturated aldehyde carbonyl was observed in addition to the band at 1728 cm\(^{-1}\) due to lactone carbonyl group. However, no twin peaks at 1386 & 1367 cm\(^{-1}\), characteristic of gemdimethyl, were observed. This was further supported by proton spectrum, wherein a down field signal at \( \delta \) 1.84 (3H, s), 1.81(3H, s) were assigned to C-3' gemdimethyl protons.

In the \( ^{13}C \) NMR spectrum, 15 carbon signals were observed. Nine carbons were assigned for a coumarin nucleus at \( \delta \) C-160.7 (-OC=O, C-2), 112.6 (=CH, C-3), 143.4 (-CH, C-4), 117.5 (C, C-4a), 126.2 (C, C-5), 112.4 (C, C-6), 159.9 (-C=O, C-7), 107.2 (C, C-8), 152.3 (C, C-8a). The rest of the other carbon signals suggested the presence of a prenyl group at \( \delta \) C-21.8 (-CH\(_2\), C-1'), 121.0 (-CH=, C-2'), 132.3 (=C, C-3'), 25.6 (CH\(_{2}\), C-3'), 17.9 (CH\(_{3}\), C-3') and 56.1 (7-methoxy).

\textbf{Antimicrobial and Antifungal activity}

Osthol and Compound 1 were bio-evaluated for their possible antimicrobial and antifungal activities (Table 1). As per the activity studies, osthol was found to be totally inactive while compound 1 showed good antibacterial activity (32 µg/ml for MRSA) as well as antifungal activity (64 µg/ml for \textit{Candida} and \textit{Aspergillus}) (Table 2).