



**Synthesis and characterization of Novel (*E*)-1-(3, 5-bis (Benzyloxy) phenyl)
-3-aryl Prop-2-en-1-ones**

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ABSTRACT

*A series of new chalcones i.e. (*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-aryl prop-2-en-1-ones (3a-o) have been synthesized by reacting 3, 5- di benzyloxy acetophenone (1) with different substituted aromatic aldehydes (2a-o) by Claisen–Schmidt condensation. All products were obtained in good yields. The structure of the compounds have been determined by elemental analysis, FT-IR, ¹H-NMR, ¹³C-NMR and mass spectral data.*

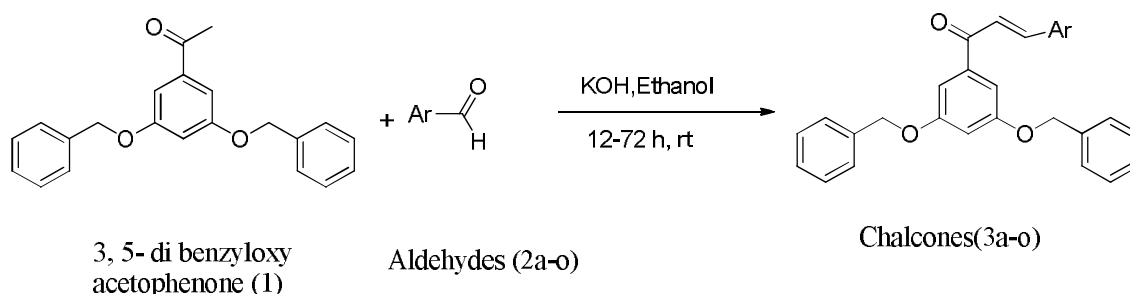
Keywords: 3, 5-dibenzyloxyacetophenone, aromatic aldehydes, Claisen–Schmidt condensation.

INTRODUCTION

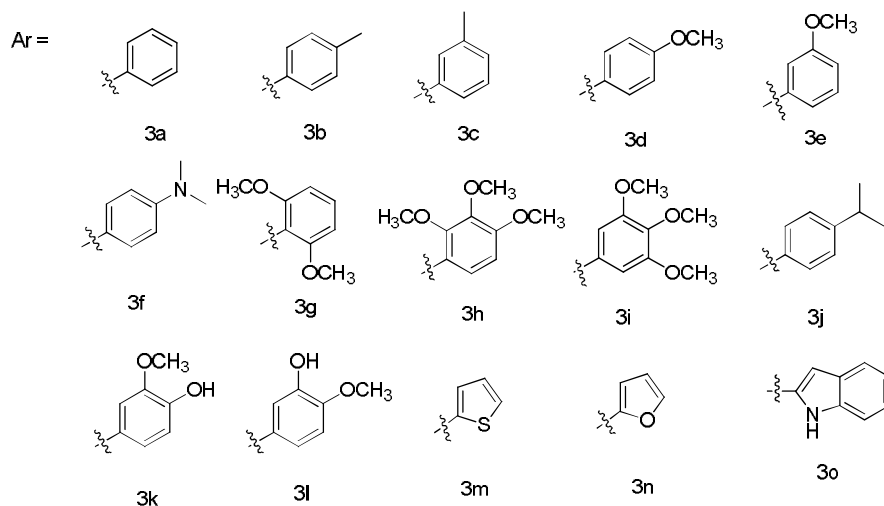
The synthesis of chalcones has always drawn the attention of chemists over the years mainly because of their important biological properties [1]. Chalcones are the main precursors for the biosynthesis of flavonoids and are present in variety of plant species such as fruits, vegetables, spices, tea and soy based foodstuff. They possess multi prolonged activities due to methylene and carbonyl moieties in their structure. Chalcones represent an important class of compounds due to their chemical flexibility, as synthons for the production of five and six membered ring systems for example Pyrazoles [2], Pyrazolines [3], isoxazolines [4], aureoles [5], pyrimidines [6, 7, 8], falvanones [9], Benzodiazepines [10] and di-aryl cyclohexenones [11]. Chalcones are known to show a broad spectrum of biological activities [12-14]. In fact, not many structural templates can claim association with such a diverse range of pharmacological activities, among which antimicrobial [15], anti-leishmanial [16], anti-malarial [17], antifungal [18], anti-viral [19], anti-inflammatory [20], cytotoxicity [21], anti-tumour [22], nematicidal [23] and anti-oxidant [24], anti-invasive [25], and anticancer [26–28] are widely cited. From the survey of literature it is observed that very few reports are available on 3, 5-bis benzyloxy phenyl chalcone derivatives. It is worthwhile to synthesise new 3, 5-bis benzyloxy phenyl chalcone derivatives. The present paper reports the synthesis, characterisation of novel chalcone derivatives (**3a-o**).

MATERIALS AND METHODS

All the chemicals were of LR grade and obtained from Avra, Sigma Aldrich and Merck. Melting points were checked on OPti-Melt automated melting point system. The purity of the compounds was checked by Thin - layer chromatography (TLC) with silica gel F254 (MERCK) visualized under UV light. The compounds are purified by column chromatography on silica gel (60-120 mesh). The instruments used for obtaining the spectroscopic data were POTIZEN3220 UV-Visible spectrophotometer, FT-IR spectrophotometer SHIMADZU-435, ^1H NMR (CDCl_3 , 400 MHz), ^{13}C NMR (CDCl_3 , INOVA 100 MHz). Mass spectral analysis using electrospray ionisation (ESI) experiments were performed using a Quadrupole time – of – flight mass spectrometer (QSTAR XL, Applied bio systems/MDS Sciex, Foster City, CA, USA) equipped with ESI source.



Scheme 1



Experimental Conditions: KOH, Ethanol, room temperature, 12-72 h

Synthesis

General Procedure for the synthesis of (E)-1-(3, 5-bis (benzyloxy) phenyl)-3- aryl prop-2-en-1-ones (3a-o): A mixture of 3, 5-di benzyloxy acetophenone (1) and substituted aromatic aldehydes (2a-o) were dissolved in ethanolic KOH. The reaction mixture was kept stirring at room temperature till the product obtained. Completion of the reaction was monitored by TLC. The residues were treated with 10% HCl in ice cold water to obtain the corresponding chalcones (3a-o) in good yield. The solid was filtered, washed with water followed by methanol. Purification of the compounds was done by column chromatography. Yields of the products varied between 48-85%.

Spectral data

Synthesis of (*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-phenylprop-2-en-1-one (3a): Yield: 0.75g (60%). m.p: 74-76°C. IR (KBr): ν_{\max} 2964, 2870, 1740, 1589, 1153 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 5.10 (s, 4H, $-\text{CH}_2\text{-O}$), 6.69 (d, 2H, $J=9.025$, Ar-H), 6.80 (t, 1H, $J=2.139$, Ar-H), 7.24 (d, 2H, $J=2.139$), 7.32-7.46 (m, 12 H, Ar-H, Ar-CH=), 7.52 (d, 2H, $J=9.025$), 8.05 (d, 1H, (β H) $J=15.560$). $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz): δ 70.36 ($-\text{CH}_2$), 106.02, 107.15, 110.95, 116.93, 122.32, 127.45, 128.05, 120.11, 130.25, 136.47, 141.00, 144.97, 151.86, 159.86, 190.02 (C=O). ESI-MS: (m/z) 421 (M+H).

(*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-(*p*-tolyl) prop-2-en-1-one (3b): Yield: 0.80g (62%). m.p: 77-79°C. IR (KBr): ν_{\max} 2986, 2821, 1741, 1592, 1154 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 2.30 (s, 3H, $-\text{CH}_3$), 5.10 (s, 4H, $-\text{CH}_2\text{-O}$) 6.47 (s, 1H, Ar-H), 7.35-7.55 (m, 17H, Ar-H, Ar-CH= (α H)), 7.84 (d, 1H, =CH (β H) $J=15.560$). ^{13}C NMR (CDCl_3 , 100 MHz) δ 22.35 ($-\text{CH}_3$), 70.16 ($-\text{CH}_2$), 106.04, 107.29, 122.35, 127.59, 128.14, 128.92, 131.94, 136.79, 137.69, 140.86, 146.10, 159.78, 189.90 (C=O). ESI-MS (m/z) 435 (M+H).

(*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-(*m*-tolyl) prop-2-en-1-one (3c): Yield: 0.79g (61%). m.p: 74-78°C. IR (KBr): ν_{\max} 2985, 2827, 1740, 1590, 1156 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 2.47 (s, 3H, $-\text{CH}_3$), 5.10 (s, 4H, $-\text{CH}_2\text{-O}$), 6.34 (s, 1H, Ar-H), 7.31-7.65 (m, 17H, Ar-H, Ar-CH=), 7.94 (d, 1H, =CH (β H) $J=15.560$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 22.14 ($-\text{CH}_3$), 70.04 ($-\text{CH}_2$), 106.04, 107.39, 122.62, 126.25, 127.68, 128.14, 128.67, 135.09, 136.75, 138.61, 146.02, 160.08, 190.38 (C=O). ESI-MS (m/z) 435 (M+H).

(*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-(4-methoxyphenyl) prop-2-en-1-one (3d): Yield 1.05g (78%). m.p: 82-85°C. IR (KBr): ν_{\max} 3029, 2836, 1740, 1588, 1152 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) : δ 3.86 (s, 3H, CH_3), 5.10 (s, 4H, $-\text{CH}_2\text{-O}$), 6.82 (t, 1H, Ar-H), 6.93 (d, 2H, $J=7.027$, Ar-H), 7.36-7.45 (m, 13H, Ar-H, Ar-CH=), 7.57 (d, 2H, Ar-H, $J=7.027$), 7.98(d, 1H, (β H) $J=15.560$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 57.19 ($-\text{OCH}_3$), 70.39 ($-\text{CH}_2$), 106.04, 107.44, 111.49, 116.19, 122.62, 127.67, 128.18, 128.97, 130.39, 136.58, 141.19, 146.04, 159.82, 190.04 (C=O). ESI-MS (m/z) 451 (M+H).

(*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-(3-methoxyphenyl) prop-2-en-1-one (3e): Yield: 0.95g (71%). m.p: 84-86°C. IR (KBr): ν_{\max} 3029, 2836, 1741, 1588, 1152 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) : δ 3.92 (s, 3H, OCH_3) 5.10 (s, 4H, $-\text{O-CH}_2$), 6.82 (s, 1H, Ar-H), 6.93 (d, 2H, Ar-H $J=8.784$), 7.20 (d 1H, Ar-H, $J=3.514$), 7.30 (d 1H, Ar-H, $J=3.514$), 7.36-7.44 (m, 11H, Ar-H, Ar-CH=), 7.58 (d, 2H, Ar-H, $J=8.784$), 7.77 (d, 1H, (β H) $J=15.560$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 57.19($-\text{OCH}_3$), 70.39 ($-\text{CH}_2$), 106.00, 107.49, 111.82, 116.19, 122.62, 127.68, 128.14, 128.87, 130.39, 136.57, 141.19, 146.05, 159.80, 190.04 (C=O). ESI-MS (m/z) 451 (M+H).

(*E*)-1-(3, 5(benzyloxy) phenyl)-3-(4-(dimethyl amino) phenyl) prop-2-en-1-one (3f): Yield: 1.18g (85%), m.p: 85-88°C. IR (KBr): ν_{\max} 2921, 2854, 1739, 1649, 1284, 1156 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 3.05 (s, 6H, $-\text{N}(\text{CH}_3)_2$), 5.10 (s, 4H, $\text{CH}_2\text{-O}$), 6.69 (d, 2H, Ar-H, $J=9.035$), 6.80 (t, 1H, Ar-H $J=2.259$), 7.24 (2H, Ar-H, $J=2.259$), 7.34-7.46 (m, 13H, Ar-H, Ar-CH=), 7.52 (d 2H, Ar-H $J=9.035$), 7.79 (d, 1H, β H $J=15.560$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 40.15 ($-\text{N}(\text{CH}_3)_2$), 70.36 ($-\text{O-CH}_2$) 106.04, 107.39, 111.82, 116.80, 122.62, 127.68, 128.14, 128.67, 130.49, 136.59, 141.17, 146.00, 152.09, 159.94, 190.04 (C=O). ESI-MS (m/z) 464 (M+H).

(*E*)-1-(3, 5-bis (benzyloxy) phenyl)-3-(2, 6-dimethoxyphenyl)-2-en-1-one (3g): Yield: 1.16g (81%). m.p: 89-91°C. IR (KBr): ν_{\max} 3025, 2866, 1740, 1571, 1157 cm^{-1} . ^1H NMR: (CDCl_3 , 400 MHz): δ 3.89 (s, 6H, 5.10 (s 4H), 6.47 (s, 1H), 6.52 (d, 1H, $J=8.784$), 6.77 (d, 2H), 7.34-7.45 (m, 11H), 7.55 (d 2H, $J=8.784$), 8.05 (d,1H $J=15.811$). ^{13}C NMR (CDCl_3 , 100 MHz) : δ 56.14 ($-\text{OCH}_3$), 70.82 ($-\text{OCH}_2$), 106.08, 107.39, 114.18, 122.68, 127.68, 128.14, 128.67, 136.59, 141.19, 146.00, 158.09, 159.34, 190.04 (C=O). ESI-MS (m/z) 481 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(2, 3, 4-trimethoxyphenyl) prop-2-en-1-one (3h): Yield: 1.13g (74%). Mp: 85-87°C. IR (KBr): ν_{\max} 2921, 2854, 1739, 1649, 1156 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 3.82 (s, 9H), 5.10 (s, 4H), 6.67 (d, 2H, $J=9.038$), 6.80 (t, 1H, $J=2.279$), 7.24 (d, 2H, $J=2.259$), 7.32-7.46 (m, 9H), 7.52 (d, 2H, $J=9.038$), 8.08 (d, 1H, $J=15.821$). $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 54.98 (-OCH₃), 59.18 (-OCH₃), 60.09 (-OCH₃), 70.95(-OCH₂), 103.91, 106.42, 107.29, 120.99, 126.59, 127.98, 128.19, 128.96, 136.80, 139.95, 147.20, 150.19, 156.02, 160.28, 190.28 (C=O). ESI-MS (m/z) 511 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(3, 4, 5-trimethoxyphenyl) prop-2-en-1-one (3i): Yield: 1.10g (72%). Mp: 83-85°C. IR (KBr): ν_{\max} 2969, 2836, 1740, 1591, 1125 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 3.84 (s, 9H), 5.10 (s, 4H), 6.68 (2H, $J=9.038$), 6.80 (t, 1H, $J=2.279$), 7.24 (d 2H, $J=2.259$), 7.38-7.46 (m, 9H), 7.53 (d, 2H, $J=9.038$), 8.06 (d, 1H, $J=15.821$). $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 54.10 (-OCH₃), 60.14 (-OCH₃), 70.36 (-OCH₂), 103.17, 106.54, 107.39, 116.80, 126.63, 127.68, 128.18, 128.67, 130.49, 136.59, 138.09, 146.02, 152.82, 159.91, 190.14 (C=O). ESI-MS (m/z) 511 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(4-isopropylphenyl) prop-2-en-1-one (3j): Yield: 0.73g (53%), m.p: 68-72°C. IR (KBr): ν_{\max} 3021, 2871, 1740, 1587, 1155 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 1.28 (d, 6H), 2.68 (septet, 1H), 5.10 (s, 4H), 6.71(d, 2H, $J=8.096$), 6.91 (t, 2H, $J=2.279$), 7.23 (d, 2H, $J=2.279$), 7.36-7.44 (m, 11H), 7.55 (d, 2H, $J=8.096$), 7.78 (d, 1H, $J=15.670$). $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 23.15 (-CH₃), 34.17 (-CH), 70.85 (-OCH₂), 106.04, 107.39, 126.67, 127.60, 128.04, 128.67, 132.60, 136.59, 136.59, 141.25, 146.14, 147.98, 160.09, 190.17 (C=O). ESI-MS (m/z) 463 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(4-hydroxy-3-methoxyphenyl) prop-2-en-1-one (3k): Yield: 0.91g (65%). m.p: 76-79°C. IR (KBr): ν_{\max} 3453, 3030, 2944, 1740, 1568, 1155 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 3.55 (s, 3H, -OCH₃), 5.09 (s, 4H, -OCH₂), 5.97-6.03 (br s, 1H, -OH), 6.89(s, 1H, Ar-H), 6.94 (d, 1H, $J=8.031$), 7.10 (s, 1H, Ar-H), 7.17 (d, 1H, Ar-H, $J=8.031$), 7.32-7.46 (m, 13H, Ar-H, Ar-CH=), 7.74 (d, 1H, $J=15.560$, =CH): $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 50.14 (-OCH₃), 70.39 (-OCH₂), 106.09, 107.39, 111.82, 116.80, 122.62, 127.68, 128.17, 128.62, 136.59, 141.28, 146.00, 147.94, 160.25, 190.12 (C=O). ESI-MS (m/z) 467 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(3-hydroxy-4-methoxyphenyl) prop-2-en-1-one (3l): Yield: 0.77g (55%). m.p: 75-79°C IR (KBr): ν_{\max} 3436, 3034, 2875, 1658, 1588, 1160 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 3.95 (s, 3H, -OCH₃), 5.09 (s, 4H, -OCH₂), 5.92-6.03 (br s 1H, -OH), 6.82 (s 1H, Ar-H), 6.94 (d, 1H, Ar-H, $J=8.031$), 7.11 (s 1H, Ar-H), 7.17 (d, 1H, Ar-H $J=8.031$), 7.31-7.45 (m, 13H, Ar-H), 7.74 (d, 1H, Ar-H, $J=15.560$). $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 56.14 (-OCH₃), 70.39 (-OCH₂), 106.09, 107.39, 111.82, 116.80, 122.62, 127.68, 128.17, 128.61, 136.59, 141.69, 146.00, 147.15, 149.52, 159.95, 190.04 (C=O). ESI-MS (m/z) 467 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(thiophen-2-yl) prop-2-en-1-one (3m): Yield: 1.02g (80%). m.p: 77-80°C ν_{\max} 3086, 2914, 1799, 1579, 1162 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 5.10 (s, 4H, -OCH₂), 6.83 (t, 1H, Ar-H $J=2.259$), 7.08 (dd, 1H); 7.22 (d, 2H, Ar-H, $J=2.259$); 7.34-7.46 (m, 13H, Ar-H, Ar-CH=), 7.92 (d, 1H, βH , $J=15.560$). $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 70.38 (-OCH₂), 106.08, 107.62, 127.61, 128.14, 128.67 130.49, 134.96, 136.59, 140.17, 159.91, 190.04 (C=O). ESI-MS (m/z) 427 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(furan-2-yl) prop-2-en-1-one (3n): Yield: 0.87g (71%). m.p: 74-76°C. IR (KBr) ν_{\max} 3030, 2945, 1740, 1588, 1152 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ 5.10 (s, 4H, -OCH₂), 6.83 (t, 1H, Ar-H), 7.09 (dd, 1H), 7.22 (d, 2H, Ar-H, $J=2.008$), 7.36-7.46 (m, Ar-H, Ar-CH=, 13H), 7.94 (d, 1H, $J=15.309$). $^{13}\text{C NMR}$ (CDCl_3 , 100 MHz): δ 70.38 (-OCH₂), 107.39, 113.80, 114.04, 127.68, 128.14, 128.59, 130.49, 136.67, 146.00, 152.09, 159.94, 190.14 (C=O). ESI-MS (m/z) 411 (M+H).

(E)-1-(3, 5-bis (benzyloxy) phenyl)-3-(1H-indol-2-yl) prop-2-en-1-one (3o): Yield: 0.62g (48%). m.p: 70-72°C. IR (KBr): ν_{\max} 3094, 3033, 2874, 1679, 1592, 1292, 1156 cm^{-1} . $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): 5.10 (s, 4H, $-\text{OCH}_2$), 6.69 (d, 2H, Ar-H $J=9.035$), 6.80 (t, 1H, Ar-H, $J=2.259$), 7.23 (d, 2H, Ar-H, $J=2.259$), 7.46-7.25 (m, 12H, Ar-H, Ar-CH=, $J=15.560$), 7.53 (d, 2H, Ar-H, $J=9.035$), 7.78 (d, 1H, $J=15.560$). ^{13}C NMR (CDCl_3 , 100 MHz): δ 70.82 ($-\text{OCH}_2$), 102.01, 106.48, 107.64, 111.82, 119.80, 122.55, 127.96, 128.27, 128.96, 130.99, 136.59, 142.09, 144.91, 160.01, 190.11 (C=O). ESI-MS (m/z) 460 (M+H).

RESULTS AND DISCUSSION

Chemistry: The reaction of 1-(3, 5-bis (benzyloxy) phenyl) ethanone (**1**) with aromatic aldehydes (**2a-o**) in the presence of ethanolic KOH resulted in the formation of chalcones (**3a-o**) in good yields (48-80 %). All compounds are yellow in colour. IR spectra of compounds showed absorption bands in the region of 3436-3453 due to Ar-OH in compounds (3k, 3l), showed absorption bands in the region of 2969-3086 cm^{-1} due to aromatic C-H stretching, 2821-2944 cm^{-1} due to aliphatic C-H stretching, 1658-1799 due to C=O functional group, 1568-1649 due to CH=CH stretching, showed absorption bands in the region of 1125-1162 due to C-O-C stretching in compounds (**3a-o**), showed absorption bands in the region of 1284-1292 due to C-N stretching in compounds (3f, 3o), indicating the evidence for the formation of titled compounds (**3a-o**). ^1H NMR spectra of compound 3j showed doublet at δ 1.28 due to $\text{CH}(\text{CH}_3)_2$ in compounds 3b, 3c showed singlet at δ 2.30-2.47 due to $-\text{CH}_3$ group, in compound 3j showed septet at 2.68 due to $\text{CH}(\text{CH}_3)_2$, in compound 3f showed singlet at δ 3.05 $-\text{N}(\text{CH}_3)_2$, in compounds 3d, 3e, 3g, 3h, 3i, 3k, 3l showed singlet at δ 3.82-3.96 due to $-\text{OCH}_3$, in compounds (**3a-o**) showed singlet at δ 5.09 - 5.10 due to $-\text{OCH}_2$, compounds (**3a-o**) showed signals in the range of δ 6.34-7.60 due to Ar-H and Ar-CO-CH= (α H) and showed doublet in the range of δ 7.75-8.08 due to (Ar-CH= (β H)). In the ^{13}C NMR spectra of compounds (3b, 3c, 3j) the chemical shift values of carbon atoms appeared at 22.43-23.15 due to (CH_3), in compound 3j at 34.17 due to $\text{CH}(\text{CH}_3)_2$, 3f at 40.15 due to $-\text{N}(\text{CH}_3)_2$, in compounds (3d, 3e, 3g, 3h, 3i) the chemical shift values of carbon atoms appeared at 54.98-60.14 due to $-\text{OCH}_3$, in compounds (**3a-o**) the chemical shift values of carbon atoms appeared at δ 70.16-70.96 due to $-\text{OCH}_2$, 190.04 (C=O). The mass spectra of compounds showed (M+H) peaks, is in agreement with their molecular formula from all spectral data. The compounds are characterised by IR, ^1H NMR, ^{13}C NMR and mass spectral data as (E)-1-(3, 5-bis (benzyloxy) phenyl)-3-aryl prop-2-en-1-ones (**3a-o**).

APPLICATIONS

Novel derivatives chalcones (**3a-o**) are synthesised, they are stable to moisture and temperature. They can be used as synthons for the production of five and six-membered heterocyclic ring systems. These compounds can be used as future drugs.

CONCLUSIONS

A series of 15 novel chalcones were synthesized i.e. (E)-1-(3, 5-bis (benzyloxy) phenyl)-3-aryl prop-2-en-1-ones (**3a-o**) and characterised by IR, ^1H NMR, ^{13}C NMR and Mass spectral data. All the products obtained were stable at room temperature.

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