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# Solvent-free synthesis, spectral correlations and antimicrobial activities of some aryl *E* 2-propen-1-ones

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# HIGHLIGHTS

# G R A P H I C A L A B S T R A C T

- Green aldol condensation gives more than 85% yield of a series of *E* 2-propen-1-ones.
- These aryl *E* 2-propen-1-ones were characterized by their analytical and spectral data.
- Hammett spectral linearity has been studied for 4'-iodophenyl chalcones.
- Antimicrobial activities of 4'-
- iodophenyl chalcones have also been studied.

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# Introduction

Numerous greener solvent-free [1,2] synthetic methods available for synthesis of organics. These solvent free reactions involving the formation of carbon-carbon bond and carbon-heteroatom bond are important and interesting in green synthesis. Based on this the Aldol [3], Crossed-Aldol [4], Knoevenagel [5], Mannich [6], Michael [7] and Wittig [8] reactions have been applied for synthesising isomeric biologically active compounds such as chalcones, alkenes

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X= H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH<sub>3</sub>, 4- OCH<sub>3</sub>, 4-CH<sub>3</sub>

# ABSTRACT

Totally 38 aryl *E* 2-propen-1-ones including nine substituted styryl 4-iodophenyl ketones have been synthesised using solvent-free SiO<sub>2</sub>–H<sub>3</sub>PO<sub>4</sub> catalyzed Aldol condensation between respective methyl ketones and substituted benzaldehydes under microwave irradiation. The yields of the ketones are more than 80%. The synthesised chalcones were characterized by their analytical, physical and spectroscopic data. The spectral frequencies of synthesised substituted styryl 4-iodophenyl ketones have been correlated with Hammett substituent constants, *F* and *R* parameters using single and multi-linear regression analysis. The antimicrobial activities of 4-iodophenyl chalcones have been studied using Bauer–Kirby method.

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and acyl compounds. Thermal condensation reactions have been found to be sluggish and time-consuming with poor yields. However in microwave conditions, the reaction is faster, giving appreciable yield involving easier process of isolation of the products. Scientists and Chemists have used microwave irradiation technique for solid phase green synthesis [8,9]. Numerous green catalysts such as flyash:sulphuric acid [1], silica-sulphuric acid [10,11] anhydrous zinc chloride [12], ground chemistry catalysts-grinding the reactants with sodium hydroxide [13], aqueous alkali in lower temperature [14], solid sulphonic acid from bamboo [15], barium hydroxide [16], anhydrous sodium bicarbonate [17], microwave assisted synthesis [18], Fly-ash:water [19], triphenylphosphite [20], alkali earth metals

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[21], KF/Al<sub>2</sub>O<sub>3</sub> [22] and sulfated titania [23] and silicotungstic acid [24] have been reported for the synthesis of many number of organic compounds. Chalcones possess various multipronged activities such as antimicrobial [25], antidepressants [26], antiplosmodial [27], anti-aids [28] and insect antifeedant activities [29]. Spectral data is useful for prediction of ground state equilibration of organic molecules such as s-cis and s-trans isomers of alkenes, alkynes, benzoyl chlorides, styrenes and  $\alpha$ ,  $\beta$ -unsaturated ketones [27,29]. The Quantitative structure activity relationship and quantitative structure property relationships were used for finding the structure of molecule, quantitative and qualitative analysis [27–29]. Their use in structure parameter correlations becomes popular for studying biological activities [30], normal co-ordinate analysis [31] and transition states of reaction mechanisms [32]. Infrared spectroscopy is a powerful tool technique for the qualitative and quantitative study of natural and synthetic molecules [33]. IR spectroscopy can provide information about the nature. concentration and structure of samples at the molecular level [34]. A great deal of work has been devoted to the reactivity of  $\alpha,\beta$ -carbonyl compounds particularly, the theoretical study of substituent effects has been studied on long range interactions in the  $\beta$ -sheet structure [35] of oligopeptides, enone-dienol tautomerism [36]. Literature study reveals QSAR study of substituted benzo[ $\alpha$ ] phenazines [37], cancer agents, Diels-Alder reactions [38], density functional theory [39], gas phase reactivity of alkyl allyl sulphides [40] and rotational barriers in selenomides [41]. Santelli et.al. [42] have studied the quantitative structural relationships in  $\alpha$ ,  $\beta$ -unsaturated carbonyl compounds between the half wave reduction potential, the frontier orbital energy and the Hammett  $\sigma_p$  values. Dhami and Stothers [43] have extensively studied the <sup>1</sup>H nmr spectra of a large number of acetophenones and styrenes with a view to establish the validity of the additivity of substituent effect in aromatic shielding first observed by Laturber [44]. Savin and co-workers [45] have studied the NMR data of unsaturated ketones of the type RC<sub>6</sub>H<sub>4-</sub> -CH=CH-COCH<sub>3</sub> and sought Hammett correlations for the ethylenic protons. Solcaniova [46] and co-workers have measured <sup>1</sup>H and <sup>13</sup>C NMR spectra of substituted styrenes, styryl phenyls and they obtained good Hammett correlations for the olefinic protons and carbons. At present, scientists [2,3,5,9,12,27] have paid more interest to correlate the group frequencies of spectral data with Hammett substituent constants to explain the substituent effects of organic compounds. Recently Thirunarayanan co-workers [2a,3,4,9,12,47-49] investigated elaborately the single and multi-regression analysis of substituent effects on alpha and beta hydrogen and carbons of some pyrrolyl, naphthyl and furyl chalcones. However there is no information available in the literature for the synthesis of chalcones using green catalyst SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> by crossed-aldol condensation, correlation study of infrared and NMR spectroscopic data with Hammett equation and antimicrobial activities in the past with substituted styryl 4-iodo phenyl ketones. Therefore the authors have taken efforts to synthesized some substituted styryl 4-iodophenyl ketones by condensation of 4-iodoacetophenone with various substituted benzaldehydes, studied the quantitative structure property relationship from the group frequencies and studied their antimicrobial activities.

#### Experimental

#### General

All chemicals used were purchased from Sigma–Aldrich and E-Merck chemical companies. Melting points of all chalcones were determined in open glass capillaries on SUNTEX melting point apparatus and are uncorrected. The UV spectra of all synthesized chalcones were recorded in ELICO-BL222 SPECTROMETER ( $\lambda_{max}$  nm) in spectral grade methanol. Infrared spectra (KBr, 4000–400 cm<sup>-1</sup>) were recorded on AVATAR-300 Fourier transform spectrophotometer. Bruker AV400 NMR spectrometer has been used for

recording NMR spectra, operating at 400 MHz has been utilized for recording <sup>1</sup>H spectra and 100 MHz for <sup>13</sup>C spectra in CDCl<sub>3</sub> solvent using TMS as internal standard. Mass spectra were recorded on a SIMADZU GC-MS2010 Spectrometer using Electron Impact (EI) techniques.

# Preparation of SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> catalyst

In a 50 mL Borosil beaker, 2 g of silica  $(10-20\mu)$  and 2 mL of ortho phosphoric acid were taken and mixed thoroughly with glass rod. This mixture was heated on a hot air oven at 85 °C for 1 h, cooled to room temperature, stored in a borosil bottle and tightly capped. This was characterized by infrared spectra and SEM analysis.

Infrared spectral data of SiO<sub>2</sub>–H<sub>3</sub>PO<sub>4</sub> are *v*(cm<sup>-1</sup>): 3437(P–OH); 2932, 2849 (P–O–H); 1747, (O=P–OH); 1091(P=O), 800(P–O); 464[50].

The SEM images of pure SiO<sub>2</sub> and SiO<sub>2</sub>–H<sub>3</sub>PO<sub>4</sub> at two different magnifications are shown in Fig. 1a–d. Fig. 1a and b depicted that the crystallinity is found to be more in SiO<sub>2</sub>. The spherical shaped particles are clearly seen at both magnifications in Fig. 1a and b. Fig. 1a reveals that the globular structure of pure silica (round shaped particle). This also seen from Fig. 1c and d that some of the particles are slightly corroded by H<sub>3</sub>PO<sub>4</sub> (indicated by arrow mark) and this may be due to dissolution of SiO<sub>2</sub>by H<sub>3</sub>PO<sub>4</sub>. This will further confirmed by Fig. 1d, the well-shaped particles of pure SiO<sub>2</sub>. Fig. 1b is aggregated to Fig. 1d due to presence of H<sub>3</sub>PO<sub>4</sub>.

# General procedure for synthesis of substituted styryl4iodophenylketones

An appropriate mixture of 4-iodoacetophenone (2 mmol) and substituted benzaldehydes (2 mmol) and  $SiO_2-H_3PO_4(0.5 g)$  taken in 50 mL corning glass tube and tightly capped. The reaction mixture was subjected to microwave irradiation at 460W for 8–10 min in a microwave oven (Scheme 1)(LG Grill, Intellowave, Microwave Oven, 160–800 W) and then cooled to room temperature. Added 10 mL of dichloromethane, the organic layer has been separated by filtration and on evaporation yields the solid product. The solid, on recrystallization with benzene-hexane mixture (1:1) afforded glittering pale yellow solid. About 10 mL of dichlorometh-ane was added, the organic layer was removed by filtration and the catalyst was recovered as solid. The catalyst was washed with ethyl acetate (10 mL) and heated at 110 °C, then it was reused for further runs.

# **Results and discussion**

In our organic chemistry research laboratory, we attempts to synthesize aryl chalcone derivatives by crossed-aldol condensation of electron withdrawing as well as electron donating group substituted acetophenone and benzaldehydes in the presence of vigorous acidic catalyst SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> in microwave irradiation. Hence the authors have synthesized the chalcone derivatives by the reaction between 2 mmol of aryl iodo ketones, 2 mmol substituted benzaldehydes in microwave irradiation with 0.5 g of SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> catalyst (Scheme 1). During the course of this reaction SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> catalyses aldol-condensation between aryl ketone and aldehydes and elimination of water gave the chalcones. The yields of the chalcones in this reaction are more than 85%. The proposed general mechanism of this reaction is given in Fig. 2. Further we have investigated this reaction with equimolar quantities of the 4-iodoacetophenone and benzaldehyde. In this reaction the obtained yield was 87%. The effect of catalyst in this reaction was studied by varying the catalyst guantity from 0.1 g to 1 g. As the catalyst quantity is increased from 0.1 g to 1 g, the percentage of yield of product is increased from 85% to



**Fig. 1.** SEM images of pure SiO<sub>2</sub> and H<sub>3</sub>PO<sub>4</sub>: (a) Pure SiO<sub>2</sub> (5  $\mu$ m); (b) pure SiO<sub>2</sub> (20  $\mu$ m); (c) SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> (5  $\mu$ m) ( $\Longrightarrow$ - corroded); (d) SiO<sub>2</sub>-H<sub>3</sub>PO<sub>4</sub> (20  $\mu$ m) ( $\Longrightarrow$ - corroded).



X= H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH<sub>3</sub>, 4- OCH<sub>3</sub>, 4-CH<sub>3</sub>

Scheme 1. Synthesis of substituted styryl 4-iodophenyl chalcones.

87%. Further increase the catalyst amount there is no significant increasing of the percentage of product. This catalytic effect is shown in (Fig. 3). The optimum quantity of catalyst loading was found to be 0.4 g. We have carried out this reaction with various substituted ketones and benzaldehydes. The results, analytical and mass spectral data are summarized in Table 1. There is no significant effect of substituents on the condensation reaction. The reusability of this catalyst was studied the aldol reaction of 4-iodoacetophenone with benzaldehyde and is presented in Table 2. From Table 2, first two runs gave 87% product. The third, fourth and fifth runs of reactions gave the yields 86.5%, 86.5% and 86% of chalcones. There was no appreciable loss in its effect of catalytic activity was observed up to fifth run. The complete spectroscopic data of selective compounds **(entries 30–38)** are summarized below.

(*E*)-1-(4-iodophenyl)-3-phenylprop-2-en-1-one(30): IR (KBr, cm<sup>-1</sup>)  $v = 1627.92(CO_{s-cis}), 1597.10(CO_{s-trans}), 1122.57(CH<sub>ip</sub>), 756.10(CH<sub>op</sub>),$ 1002.98(CH=CH<sub>op</sub>), 551.64(C=C<sub>op</sub>); <sup>1</sup>H NMR(400 MHz, CDCl<sub>3</sub>, $ppm): <math>\delta = 7.740(1H, \alpha, d), 7.877(1H, \beta, d), 7.263-7.867(9H, m, Ar-H);$  <sup>13</sup>C NMR(100 MHz CDCl<sub>3</sub>, ppm):  $\delta = 121.48(C_{\alpha}), 145.47(C_{\beta}),$ 189.75(CO), 137.50(C<sub>1</sub>), 129.96(C<sub>2</sub>, C<sub>6</sub>), 144.03(C<sub>3</sub>, C<sub>5</sub>), 100.63(C<sub>4</sub>), 134.60(C'<sub>1</sub>), 128.55(C'<sub>2</sub>, C'<sub>6</sub>), 130.81(C'<sub>3</sub>, C'<sub>5</sub>), 121.48(C'<sub>4</sub>).

(*E*)-1-(4-iodophenyl)-3-(4-bromophenyl)prop-2-en-one(31): IR (KBr, cm<sup>-1</sup>)  $\nu$  = 1654.72(CO<sub>s-cis</sub>), 1604.77(CO<sub>s-trans</sub>), 1103.28(CH<sub>ip</sub>), 798.53(CH<sub>op</sub>), 1010.70(CH=CH<sub>op</sub>), 543.93(C=C<sub>op</sub>); <sup>1</sup>H NMR(400 MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 7.564(1H,  $\alpha$ , d), 7.878(1H,  $\beta$ , d), 7.264–7.768(8H m, Ar-H); <sup>13</sup>C NMR(100MHz, CDCl<sub>3</sub>, ppm):  $\delta$  = 121.90(C $\alpha$ ),  $\begin{array}{l} 144.00(C_{\beta}), \ 189.44(CO), \ 137.28(C_{1}), \ 132.31(C_{2}, \ C_{6}), \ 138.03(C_{3}, \ C_{5}), \\ 100.20(C_{4}), \ 133.62(C_{1}'), \ 129.89(C_{2}', C_{6}'), \ 129.93(C_{3}', C_{5}'), \ 121.90(C_{4}'). \end{array}$ 

 $\begin{array}{l} (E)-1-(4-iodophenyl)-3-(4-chlorophenyl)prop-2-en-1-one(32):\\ \mathrm{IR}(\mathrm{KBr},\ \mathrm{cm}^1)\ v=1666.50(\mathrm{CO}_{s-cis}),\ 1598.31(\mathrm{CO}_{s-trans}),\ 1105.21(\mathrm{CH}_{ip}),\\ 831.32(\mathrm{CH}_{op}),\ 1056.99(\mathrm{CH=CH}_{op}),\ 536.21(\mathrm{C=C}_{op});\ ^{1}\mathrm{H}\ \mathrm{NMR}(400\mathrm{MHz},\ \mathrm{CDCl}_3,\ \mathrm{ppm})\ \delta=7.579(1\mathrm{H},\ \alpha,\ d),\ 7.879(1\mathrm{H},\ \beta,d),\ 7.264-7.787(8\mathrm{H},\ m,\ \mathrm{Ar-H});\ ^{13}\mathrm{C}\ \mathrm{NMR}(\mathrm{CDCl}_3,\ \mathrm{ppm});\ \delta=121.81(\mathrm{C}\alpha),\ 143.95(\mathrm{C}\beta),\ 189.25(\mathrm{CO});\ 138.02(\mathrm{C}_1),\ 129.35(\mathrm{C}_2,\mathrm{C}_6),\ 129.70(\mathrm{C}_3,\mathrm{C}_5),\ 100.48(\mathrm{C}_4),\ 133.19(\mathrm{C}_1'),\ 127.44(\mathrm{C}_2',\mathrm{C}_6'),\ 138.02(\mathrm{C}_3',\ \mathrm{C}_5'),\ 129.93(\mathrm{C}_4'). \end{array}$ 

 $\begin{array}{l} (E)-1-(4-iodophenyl)-3-(2-fluorophenyl)prop-2-en-1-one(33):\\ \mathrm{IR}(\mathrm{KBr},\ \mathrm{cm}^{-1}) \quad v=1660.71(\mathrm{CO}_{s-cis}),\ 1602.90(\mathrm{CO}_{s-trans}),\ 1095.60\\ (\mathrm{CH}_{ip}),\ 813.96(\mathrm{CH}_{op}),\ 1002.98(\mathrm{CH=CH}_{op}),\ 511.14(\mathrm{C=C}_{op});\ ^{1}\mathrm{H}\\ \mathrm{NMR}(400\ \mathrm{MHz},\ \mathrm{CDCl}_3,\ \mathrm{ppm}) \quad \delta=7.745(1\mathrm{H},\ \alpha,\ d),\ 7.883(1\mathrm{H},\ \beta,\ d),\\ 7.127-7.928(8\mathrm{H},\ m,\ \mathrm{Ar-H});\ ^{13}\mathrm{C}\ \mathrm{NMR}(100\ \mathrm{MHz},\ \mathrm{CDCl}_3,\ \mathrm{ppm}) \\ \delta=122.68(\mathrm{C}\alpha),\ 138.00(\mathrm{C}\beta),\ 189.61(\mathrm{CO}),\ 137.20(\mathrm{C}_1),\ 132.09(\mathrm{C}_2),\\ 137.95(\mathrm{C}_3),\ 100.85(\mathrm{C}_4),\ 138.00(\mathrm{C}_5),\ 132.18(\mathrm{C}_6),\ 123.84(\mathrm{C}_1),\\ 160.46(\mathrm{C}_2'),\ 116.21(\mathrm{C}_3'),\ 129.77(\mathrm{C}_4'),\ 124.58(\mathrm{C}_5'),\ 129.80(\mathrm{C}_6'). \end{array}$ 

 $\begin{array}{ll} (E)-1-(4-iodophenyl)-3-(4-fluorophenyl)prop-2-en-1-one(34):\\ \mathrm{IR}(\mathrm{KBr},\ \mathrm{cm}^{-1}) \quad \nu=1654.92(\mathrm{CO}_{s-cris}),\ 1593.20(\mathrm{CO}_{s-trans}),\ 1103.31\\ (\mathrm{CH}_{ip}),\ 823.60(\mathrm{CH}_{op}),\ 987.55(\mathrm{CH=CH}_{op}),\ 514.99(\mathrm{C=C}_{op}),\ ^1\mathrm{H}\\ \mathrm{NMR}(400\ \mathrm{MHz}\ \mathrm{CDCl}_3,\ \mathrm{ppm}) \quad \delta=7.393(1\mathrm{H},\ \alpha,\ d),\ 7.78(1\mathrm{H},\ \beta,\ d),\\ 7.096-7.801(8\mathrm{H},\ m,\ \mathrm{Ar-H});\ ^{13}\mathrm{C}\ \mathrm{NMR}(100\ \mathrm{MHz},\ \mathrm{CDCl}_3,\ \mathrm{ppm}) \\ \delta=121.14(\mathrm{C}_{\alpha}),\ 144.13(\mathrm{C}_{\beta}),189.50(\mathrm{CO}),\ 137.39(\mathrm{C}_1),\ 130.99(\mathrm{C}_2,\mathrm{C}_6),\\ 137.99(\mathrm{C}_3,\mathrm{C}_5),\ 100.75(\mathrm{C}_4),\ 130.95\ (\mathrm{C}_1'),\ 129.92\ (\mathrm{C}_2',\mathrm{C}_6'),\ 116.24\\ (\mathrm{C}_3',\mathrm{C}_5'),\ 162.96\ (\mathrm{C}_4'). \end{array}$ 



(entry 30; X=H)

Fig. 2. Synthesis of substituted styryl 4-iodophenyl ketones by silica-phosphoric acid catalyzed aldol condensation.



Fig. 3. Effect of catalyst loading.

(*E*)-1-(4-iodophenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one(35): IR(KBr, cm<sup>-1</sup>)  $v = 1670.35(CO_{s-cris})$ , 1578.00(CO<sub>s-trans</sub>), 1111.00 (CH<sub>ip</sub>), 815.89(CH<sub>op</sub>), 1004.91(CH=CH<sub>op</sub>), 592.15(C=C<sub>op</sub>); <sup>1</sup>H NMR(400 MHz, CDCl<sub>3</sub>, ppm)  $\delta = 7.668(1H, \alpha, d)$ , 7.837(1H,  $\beta, d$ ), 7.124–7.839(8H, m, Ar-H), 4.867(1H, s, -OH); <sup>13</sup>C NMR(100 MHz, CDCl<sub>3</sub>, ppm)  $\delta = 121.81(C_{\alpha})$ , 143.95(C<sub>β</sub>), 189.25(CO), 137.94(C<sub>1</sub>), 129.76(C<sub>2</sub>, C<sub>6</sub>), 144.03(C<sub>3</sub>, C<sub>5</sub>), 101.15(C<sub>4</sub>), 134.60(C'<sub>1</sub>), 128.55 (C'<sub>2</sub>, C'<sub>6</sub>), 130.81(C'<sub>3</sub>, C'<sub>5</sub>), 143.48(C'<sub>4</sub>).

(*E*)-1-(4-iodophenyl)-3-(2-methoxyphenyl)prop-2-en-1-one(36): IR(KBr,cm<sup>-1</sup>)  $v = 1660.71(CO_{s-cis}), 1589.34(CO_{s-trans}), 1109.50(CH<sub>i</sub>p), 815.90(C-H<sub>o</sub>p), 1008.77(CH=CH<sub>o</sub>p), 534.328(C=C<sub>o</sub>p); <sup>1</sup>H NMR (400 MHz CDCl<sub>3</sub>, ppm) <math>\delta = 7.554(1H, \alpha, d), 8.113(1H, \beta, d), 6.943-7.851(8H, m, Ar-H), 3.923(s, 3H, OCH<sub>3</sub>); <sup>13</sup>C NMR(100 MHz, CDCl<sub>3</sub>, methods)$  ppm)  $\delta = 120.81(C\alpha), 141.09(C\beta), 190.41(CO), 137.86(C_1), 132.04$ (C<sub>2</sub>), 139.74(C<sub>3</sub>, C<sub>5</sub>), 100.32(C<sub>4</sub>), 111.28(C'\_1, C'\_3), 158.90(C'\_2), 122.31(C'\_5), 129.40(C'\_4), 130.03(C'\_6).

(*E*)-1-(4-iodophenyl)-3-(4-methoxyphenyl)prop-2-en-1-one(37): IR(KBr, cm<sup>-1</sup>)  $v = 1640.37(CO_{s-cis})$ , 1593.22( $CO_{s-trans}$ ), 1134.10 (CH<sub>ip</sub>), 812.03(CH<sub>op</sub>), 1006.84(CH=CH<sub>op</sub>), 524.64(C=C<sub>op</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, ppm)  $\delta = 7.425(1H, \alpha, d)$ , 7.799(1H,  $\beta, d$ ), 7.224– 7.815(8H, m, Ar-H), 3.864(3H, s,-OCH<sub>3</sub>); <sup>13</sup>C NMR(100 MHz, CDCl<sub>3</sub>, ppm)  $\delta = 120.45(C_{\alpha})$ , 145.59( $C_{\beta}$ ), 189.33(CO), 137.65(C<sub>1</sub>), 129.94 (C<sub>2</sub>, C<sub>6</sub>), 131.98(C<sub>3</sub>, C<sub>5</sub>), 100.48(C<sub>4</sub>), 128.60(C'<sub>1</sub>), 128.60(C'<sub>2</sub>, C'<sub>6</sub>), 137.88(C'<sub>3</sub>, C'<sub>5</sub>), 145.59(C'<sub>4</sub>).

 $\begin{array}{ll} (E)-1-(4-iodophenyl)-3-tolylphenyl-prop-2-en-1-one(38): \mbox{ IR(KBr,} (cm^{-1}) & v = 1653(CO_{s-cis}), & 1591.30(CO_{s-trans}), & 1114.90(CH_{ip}), \\ 815.89(CH_{op}), & 1018.41(CH=CH_{op}), & 530.42(C=C_{op}); & ^1H & NMR \\ (400 \ MHz \ CDCl_3, \ ppm) & \delta = 6.943(1H \ \alpha, \ d), & 7.604(1H, \ \beta, \ d), & 2.401(s, \\ 3H, \ CH_3,); & ^{13}C & NMR(100 \ MHz \ CDCl_3, \ ppm) & \delta = 119.12(C_{\alpha}), \\ 145.33(C_{\beta}), & 189.74(CO), & 137.88(C_1), & 129.89(C_2, \ C_6), & 130.38(C_3, \ C_5), \\ 100.18(C_4), & 134.60(C_1'), & 127.44(C_2', C_6'), & 137.88(C_3', C_5'), & 130.13(C_4'). \end{array}$ 

# Spectral linearity

In the present study the spectral linearity of chalcones has been studied by evaluating the substituent effects [2a,3,4,9,12,27,29,47–50] on the group frequencies. The assigned group frequencies of all chalcones line infrared carbonyl stretches  $vCO_{s-cis}$  and s-trans, the deformation modes of vinyl part CH<sub>out of plane</sub>, in-plane, CH=CH and  $>C=C<_{out of planes}$  (cm<sup>-1</sup>), NMR chemical shifts  $\delta$ (ppm) of H<sub> $\alpha$ </sub>, H<sub> $\beta$ </sub>, C<sub> $\alpha$ </sub>, C<sub> $\beta$ </sub>, CO are assigned and these frequencies are correlated with various substituent constants.

## UV-Vis and IR spectral study

The measured absorption maxima ( $\lambda_{max}$  nm) of these chalcones are presented in Table 3. These values are correlated with Hammett substituent constants and *F* and *R* parameters using single Table 1

Analytical and mass spectral data of chalcones synthesized by  $SiO_2-H_3PO_4$  catalyzed aryl methyl ketones and substituted benzaldehydes reaction of the type  $ArCOCH_3 + Ar'CHO \rightarrow ArCOCH=CHAr'$  under microwave irradiation.

Entry	/ Ar	Ar'	Product	MW	Yield (%)	M.p. (°C)	Mass $(m/z)$
1	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	208	86	55-56 (55-58)[1]	-
2	C <sub>6</sub> H <sub>5</sub>	4-ClC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> Cl	242	86	114-115	-
3	C <sub>6</sub> H <sub>5</sub>	C <sub>10</sub> H7(1- Naph)	C <sub>6</sub> H <sub>5</sub> COCH=CHC <sub>10</sub> H <sub>7</sub>	273	85	(112–114) [1] 104–105	-
4	C <sub>6</sub> H <sub>5</sub>	4-OHC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> OH	291	84	(104-105) [1] 122–123 (122) [1]	-
5	C <sub>6</sub> H <sub>5</sub>	$4-OCH_3C_6H_4$	C <sub>6</sub> H <sub>5</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> OCH <sub>3</sub>	291	84	(122) [1] 164-165 (164) [1]	-
6	$4-NH_2C_6H_5$	C <sub>4</sub> H <sub>3</sub> (3-Furyl)	) 4-NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>4</sub> H <sub>3</sub> O	213	82	120–121 (119–120) [1]	-
7	$4-NH_2C_6H_4$	$C_6H_5$	4-NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	223	82	98–99 (98–99) [1]	-
8	$4-NH_2C_6H_4$	4- N(C <sub>2</sub> H <sub>5</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>2</sub>	4- 4 NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> N(C <sub>2</sub> H <sub>5</sub> );	294 2	82	90-91	-
9	$4-NH_2C_6H_4$	2,6-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	4-NH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>3</sub> Cl <sub>2</sub>	291	80	(90–91) [1] 98–99 (98–90) [1]	-
10	4-ClC <sub>6</sub> H₄ C <sub>6</sub> H₅ 4- ClC <sub>6</sub> H₄COCH=CHC <sub>6</sub> H₅ 242				83	99–100	-
11	4-F-C <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4-FC <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	226	83	(98–100) [1] 49–50	-
12	2, 4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>	2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	276	84	(49–50) [1] 80–82	-
13	3, 4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	C <sub>6</sub> H <sub>5</sub>	3,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	276	84	(80–81)[1] 100–101	_
14	4-(OH)C <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4-(OH)C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	226	84	(100–101) [1] 63–64	-
15	C <sub>10</sub> H <sub>7</sub>	C <sub>6</sub> H <sub>5</sub>	C <sub>10</sub> H <sub>7</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	258	84	(63–64) [1] 100–102	-
16	(1-Naph) 4-BrC <sub>10</sub> H <sub>6</sub>	C <sub>6</sub> H <sub>5</sub>	4-BrC <sub>10</sub> H <sub>6</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	396	55	(100–102) [1] 103–104 (102–104) [1]	-
17	(1-Naph) 4-ClC <sub>10</sub> H <sub>6</sub> (1-Naph)	C <sub>6</sub> H <sub>5</sub>	4-CIC <sub>10</sub> H <sub>6</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	292	44	(103-104) [1] 122-123 (122-123) [1]	-
18	(1-Naph) 4-OCH <sub>3</sub> C <sub>10</sub> H <sub>6</sub> (1-Naph)	$C_6H_5$	4-OCH <sub>3</sub> C <sub>10</sub> H <sub>6</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	310	83	(122-123)[1] 113-114 (112-114)[1]	-
19	(1-Naph) 4-CH <sub>3</sub> C <sub>10</sub> H <sub>6</sub> (1-Naph)	$C_6H_5$	4-CH <sub>3</sub> C <sub>10</sub> H <sub>6</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	284	84	(113-114) [1] 98-99 (98) [1]	-
20	$C_{10}H_7$ (2-Naph)	$C_6H_5$	C <sub>10</sub> H <sub>7</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	258	84	(30) [1] 104–105 (104–105) [1]	-
21	$6-OCH_3C_{10}H_6$ (2-Naph)	C <sub>6</sub> H <sub>5</sub>	6-OCH <sub>3</sub> C <sub>10</sub> H <sub>6</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	310	85	67–68 (67–68) [1]	-
22	$6-CH_3C_{10}H_6$ (2-Naph)	$C_6H_5$	6-CH <sub>3</sub> C <sub>10</sub> H <sub>6</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	284	84	123–124 (123–124) [1]	-
23	C <sub>13</sub> H <sub>9</sub> (2-Fluorene)	$C_6H_5$	C <sub>13</sub> H <sub>9</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	296	80	150–151 (150–151) [1]	-
24	C <sub>12</sub> H <sub>9</sub> (Biphenyl)	$C_6H_5$	C <sub>12</sub> H <sub>9</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	284	85	153–154 (153–154) [1]	-
25	$C_4H_3O(2$ -Furyl)	$C_6H_5$	C <sub>4</sub> H <sub>3</sub> OCOCH=CHC <sub>6</sub> H <sub>5</sub>	198	85	80–81 (80–81) [1]	-
26	5-CH <sub>3</sub> C <sub>4</sub> H <sub>2</sub> N (2-Pyrrole)	$C_6H_5$	5-CH <sub>3</sub> C <sub>4</sub> H <sub>2</sub> NCOCH=CHC <sub>6</sub> H <sub>5</sub>	210	84	137–138 (137–138) [1]	-
27	$C_4H_3S(2-Thienyl)$	$C_6H_5$	C <sub>4</sub> H <sub>3</sub> SCOCH=CHC <sub>6</sub> H <sub>5</sub>	204	85	112–113 (112–113) [1]	-
28	$C_{14}H_9(Anthracene)$	C <sub>6</sub> H <sub>5</sub>	C <sub>14</sub> H <sub>9</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	308	82	124–125 (124–125) [1]	-
29	5-BrC <sub>4</sub> H <sub>2</sub> S (2-Thienyl)	C <sub>6</sub> H <sub>5</sub>	5-BrC <sub>4</sub> H <sub>2</sub> SCOCH=CHC <sub>6</sub> H <sub>5</sub>	292	86	107–110 (106–107) [1]	-
30	4-IC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4-I-C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>5</sub>	334	87	86–87[51]	334[M+], 256, 230, 207, 203, 131,103,77
31	4-IC <sub>6</sub> H <sub>4</sub>	4-BrC <sub>6</sub> H <sub>4</sub>	4-I-C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> Br-4	413	86	173–174	413[M+], 415[M <sup>2</sup> +], 285, 257, 231, 209, 203, 181, 155,126.
32 33	4-IC <sub>6</sub> H <sub>4</sub> 4-IC <sub>6</sub> H <sub>4</sub>	4-CIC <sub>6</sub> H₄ 2-FC <sub>6</sub> H₄	$4-I-C_6H_4COCH=CHC_6H_4CI-4$ $4-I-C_6H_4COCH=CHC_6H_4F-2$	369 352	80 85	108–109 72–74	241, 231, 209, 203, 181, 155,127 352[M+], 257, 231, 225, 203, 127, 121.
34	$4-IC_6H_4$	$4-FC_6H_4$	4-I-C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> F-4	352	80	111-112	352[M+], 257, 231, 225, 203, 127,121.
35 36	$\begin{array}{l} 4\text{-IC}_6\text{H}_4\\ 4\text{-IC}_6\text{H}_4 \end{array}$	4-OHC <sub>6</sub> H <sub>4</sub> 2-OCH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	$\begin{array}{l} \text{4-I-C}_6\text{H}_4\text{COCH} = \text{CHC}_6\text{H}_4\text{OH-4} \\ \text{4-I-C}_6\text{H}_4\text{COCH} = \text{CHC}_6\text{H}_4\text{OCH}_3\text{-2} \end{array}$	350 364	84 85	98–99 73–74	350[M+], 257, 231, 223, 203, 147, 93. 364[M+], 257, 237, 231, 203, 161, 133, 129, 107

#### Table 1 (continued)

Entry Ar	Ar′	Product	MW Yield (%)	M.p. (°C)	Mass (m/z)
37 4-IC <sub>6</sub> H <sub>4</sub>	4-OCH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	4-I-C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> OCH <sub>3</sub> -4	364 88	78–79[52]	364[M+], 257, 237, 231, 203, 161, 133, 129,107
38 4-IC <sub>6</sub> H <sub>4</sub>	$4-CH_3C_6H_4$	4-I-C <sub>6</sub> H <sub>4</sub> COCH=CHC <sub>6</sub> H <sub>4</sub> CH <sub>3</sub> -4	348 80	135–136	348[M+], 257, 231, 221, 203, 145, 127, 117, 91

#### Table 2

Reusability of catalyst on condensation of 4-iodoacetophenone(2 mmol) and benzaldehydes (2 mmol) under microwave irradiation (entries **30-38**).

Run	1	2	3	4	5
Yield	87	87	86.5	86.5	86

and multi-linear regression analysis [11,12,26–29]. Hammett correlation involving the group frequencies and absorption maxima, the form of the Hammett equation employed is

$$\lambda = \rho \sigma + \lambda_0 \tag{1}$$

where  $\lambda_o$  is the frequency for the parent member of the series.

The results of statistical analysis[2,3,5,9,12,27–29,47–50] of these values with Hammett substituent constants, *F* and *R* parameters, the Hammett  $\sigma$  constants correlated satisfactorily with r = 0.969. The remaining parameters were failing in the correlation. This is due to the polar, filed, resonance and inductive effects of the substituents were weak for predicting the reactivity on the absorption. This is evident with resonance conjugative structure shown in Fig. 4. The multi regression analysis of these frequencies of all ketones with inductive, resonance and Swain–Lupton's [53] constants produce satisfactory correlations as evident in

$$\lambda_{\max}^{(nm)} = 300.506(\pm 15.572) + 40.321(\pm 14.968)\sigma_{I} - 6.259(\pm 4.557)\sigma_{R} \quad (R = 0.942, n = 9, P > 90\%)$$
(2)

$$\begin{split} \lambda_{\max}^{(nm)} &= 300.086(\pm 15.911) + 12.221(\pm 3.362)F \\ &\quad -25.691(\pm 4.469)R \quad (R) \\ &\quad = 0.939, n = 9, P > 90\% \end{split} \tag{3}$$

IR spectral study

The carbonyl stretching frequencies  $(cm^{-1})$  of *s*-*cis* and *s*-*trans* isomers of present study are presented in Table 3 and the corresponding conformers are shown in Fig. 5. The stretching frequencies for carbonyl absorption are assigned based on the assignments made by Hays and Timmons [54] for *s*-*cis* and *s*-*trans* conformers at 1690 and 1670 cm<sup>-1</sup>, respectively.



Fig. 4. The resonance-conjugative structure.

These data have been correlated with Hammett substituent constants and Swain–Lupton constants [53] (see Table 4.). In this correlation the structure parameter Hammett equation employed is as shown in the following equation:

$$v = \rho \sigma + v_0 \tag{4}$$

where v is the carbonyl frequencies of substituted system and  $v_0$  is the corresponding quantity of unsubstitued system;  $\sigma$  is a Hammett substituent constant, which in principle is characteristics of the substituent and  $\rho$  is a reaction constant which is depend upon the nature of the reaction.

The results of single parameter statistical analysis of carbonyl frequencies with Hammett  $\sigma$  constants, *F* and *R* parameters gave poor correlations (see Table 4). A satisfactory correlation was found for *s*-trans conformers with  $\sigma$ , (r = 0.983) and  $\sigma^+(r = 0.987)$  values. The remaining parameters were fail in correlations. The failure in correlation is due the conjugation between the substituent and the carbonyl group in chalcones as shown in Fig. 4.

A satisfactory correlation obtained for CH*in-plane* modes with Hammett  $\sigma$  (r = 0.956),  $\sigma$ +(r = 0.946),  $\sigma_l$ (r = 0.963), constants and F (r = 0.968) parameter. The remaining Hammett substituent constants gave poor correlation due to the conjugation. A satisfactory correlation was found for CH*out-plane* modes with Hammett  $\sigma_R$ (r = 0.900), constant and R (r = 0.900) parameter. The remaining parameters were fail in correlations. A satisfactory correlation obtained for CH=CH *out of plane* with Hammett  $\sigma_R$  constant, F and Rparameters gave satisfactory correlation co-efficients r = 0.900, r = 0.902 and r = 0.900. The C=C *out of plane* modes with Hammett  $\sigma_R$  constant, F and R parameter correlation is satisfactorily with the

#### Table 3

The UV ( $\lambda_{max}$ , nm) absorption and IR ( $\nu$ , cm<sup>-1</sup>) spectroscopic data of substituted styryl 4-iodophenyl ketones (entries 30–38).

Entry	Х	$\lambda_{\max}$	CO <sub>s-cis</sub>	CO <sub>s-trans</sub>	CH <sub>ip</sub>	CHop	CH=CH <sub>op</sub>	C=C <sub>op</sub>
30	Н	318	1627.92	1597.10	1122.57	756.10	1002.98	551.64
31	4-Br	321	1654.72	1604.77	1103.28	798.53	1010.70	543.93
32	4-Cl	320	1666.50	1598.31	1105.21	813.32	1056.99	536.21
33	2-F	312	1660.71	1602.90	1095.60	813.96	1002.98	511.14
34	4-F	316	1654.92	1593.20	1103.31	823.60	987.55	514.99
35	4-OH	349	1670.35	1578.00	1111.00	815.89	1004.91	592.15
36	2-OCH <sub>3</sub>	301	1660.71	1589.34	1109.50	815.90	1008.77	534.28
37	4-OCH <sub>3</sub>	327	1640.37	1593.22	1134.10	812.03	1006.84	524.64
38	4-CH <sub>3</sub>	267	1653.01	1591.33	1114.90	815.89	1018.41	530.42



Fig. 5. The s-cis and s-trans conformers of substituted styryl 4-iodophenyl ketones.

#### Table 4

.

Results of statistical analysis of infrared  $v(cm^{-1}) CO_{s-cis}$ ,  $CO_{s-trans}$ ,  $CH_{ip}$ ,  $CH=CH_{op}$  and  $C=C_{op}$  substituted styryl 4-iodophenyl ketones (**entries 30–38**) with Hammett  $\sigma$ ,  $\sigma^*$ ,  $\sigma_I$ ,  $\sigma_R$  constants and F and R parameters.

Frequency	Constants	r	Ι	ρ	S	n	Correlated derivatives
CO <sub>s-cis</sub>	σ	0.745	1654.36	0.173	14.04	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.801	1654.26	-0.474	14.04	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_I$	0.811	1644.51	32.120	11.97	9	H, 4-Br, 4-Cl,2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.751	1645.25	-29.069	12.42	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.801	1645.69	23.857	12.30	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.855	1643.88	-26.117	12.16	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
CO <sub>s-trans</sub>	σ	0.983	1595.23	26.568	4.68	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.987	1596.95	13.411	4.12	8	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_I$	0.833	1590.31	12.677	8.08	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.798	1598.37	13.315	8.04	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.825	1590.97	8.888	8.21	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.752	1600.23	15.033	7.57	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
CH <sub>ip</sub>	$\sigma$	0.956	1110.03	-25.979	10.21	7	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.964	1108.12	-14.198	9.50	7	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_{I}$	0.963	1121.68	-34.689	9.54	8	H, 4-Br, 4-Cl,2-F, 4-F,4-OH, 2-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.825	1114.83	12.084	12.10	9	H, 4-Br, 4-Cl,2-F, 4-F,4-OH,2 -OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.968	1121.92	-29.951	9.04	8	H, 4-Br, 4-Cl, 2-F, 4-F,4-OH, 2-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.817	1114.25	7.995	12.22	9	H, 4-Br, 4-Cl, 2-F, 4-F,4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
CHop	$\sigma$	0.815	806.75	-12.656	21.40	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.825	805.21	-9.905	20.95	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_{I}$	0.745	793.27	45.556	19.01	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.900	787.51	-62.995	16.36	8	4-Br, 4-Cl,2-F, 4-F,4-OH,2-OCH <sub>3</sub> ,4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.805	792.71	40.010	18.46	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.900	784.89	-55.732	15.66	8	4-Br, 4-Cl,2-F, 4-F,4-OH,2-OCH <sub>3</sub> ,4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
CH=CH <sub>op</sub>	$\sigma$	0.824	1011.92	20.372	19.62	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.802	1011.82	3.394	20.29	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_{I}$	0.800	1010.97	0.513	20.38	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.900	1022.00	34.706	18.81	8	H, 4-Br, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.902	1016.48	-14.748	19.95	8	H, 4-Br, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.900	1024.73	33.928	18.22	8	H, 4-Br, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
$C = C_{op}$	$\sigma$	0.801	536.14	-40.339	23.36	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.804	533.63	-19.863	23.25	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_I$	0.824	548.45	-35.022	24.52	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.900	543.98	20.028	25.39	8	H, 4-Br, 4-Cl, 2-F, 4-F,4-OH, 2-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.900	551.38	-37.645	23.47	8	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.900	538.45	1.855	25.78	8	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-CH <sub>3</sub>

*r* = correlation coefficient;  $\rho$  = slope; *I* = intercept; *s* = standard deviation; *n* = number of substituents.

r values of 0.900, 0.900, 0.900 respectively. The remaining Hammett substituent constants gave poor correlation due to the conjugation between the substituent and the vinyl group in chalcones as shown in Fig. 5.

In view of the inability of some of the  $\sigma$  constants to produce individually satisfactory correlations, it was thought that worthwhile to seek multiple correlations involving either  $\sigma_I$  and  $\sigma_R$  constants or Swain–Lupton's [53], *F* and *R* parameters. The correlation equations for *s*-*cis*, *s*-*trans* and *deformation modes* are given in

$$v \text{CO}_{\text{s-cis}}^{(\text{cm}^{-1})} = 1642.023(\pm 8.451) - 23.500(\pm 4.403)\sigma_{\text{I}} - 16.356(\pm 4.723)\sigma_{\text{R}} - 16.356(\pm 4.723)\sigma_{\text{R}} - (R = 0.956, n = 9, P > 95\%)$$
(5)

$$v \text{CO}_{s\text{-}cis}^{(\text{cm}^{-1})} = 1642.235(\pm 8.628) + 14.240(\pm 2.803)F \\ - 17.316(\pm 2.946)R \\ (R = 0.955, n = 9, P > 95\%)$$
 (6)

(7)

(8)

$$\begin{split} v\text{CO}_{s\text{-trans}}^{(\text{cm}^{-1})} &= 1594.593(\pm 4.406) + 27.548(\pm 12.725)\sigma_{I} \\ &\quad + 28.217(\pm 12.891)\sigma_{R} \\ &\quad (R = 0.971, \ n = 9, \ P > 95\%) \end{split}$$

$$vCO_{s-trans}^{(cm^{-1})} = 1597.200(\pm 3.374) + 26.243(\pm 8.135)F + 31.251(\pm 8.582)R (R = 0.984, n = 9, P > 95\%)$$

$$vCH_{ip}^{(cm-1)} = 1120.261(\pm 6.852) - 39.614(\pm 9.787)\sigma_{I} - 9.345(\pm 2.046)\sigma_{R} (R = 0.965, n = 9, P > 95\%)$$
(9)

$$vCH_{ip}^{(cm-1)} = 1118.732(\pm 6.143) - 38.842(\pm 14.812)F + 16.010(\pm 5.625)R (R = 0.974, n = 9, P > 95\%)$$
(10)

$$vCH_{op}^{(cm-1)} = 785.136(\pm 11.710) + 17.281(\pm 13.814)\sigma_{I} - 53.646(\pm 34.257)\sigma_{R} (R = 0.967, n = 9, P > 95\%)$$
(11)

$$vCH_{op}^{(cm^{-1})} = 783.298(\pm 11.295) + 13.794(\pm 7.233)F - 47.207(\pm 28.728)R (R = 0.970, n = 9, P > 95\%)$$
(12)

$$\begin{split} \nu \text{CH} = & \text{CH}_{op}^{(\text{cm}^{-1})} = 1018.391(\pm 13.247) + 26.304(\pm 18.253)\sigma_l \\ & + 48.935(\pm 18.754)\sigma_R \\ & (R = 0.945, \ n = 9, \ P > 90\%) \end{split} \tag{13}$$

$$vCH = CH_{op}^{(cm^{-1})} = 1024.015(\pm 13.383) + 6.232(\pm 2.267)F + 7.779(\pm 34.039)R (R = 0.945, n = 9, P > 90\%)$$
(14)

$$vC = C_{op}^{(cm^{-1})} = 548.681(\pm 17.927) - 34.223(\pm 11.767)\sigma_{I} + 51.515(\pm 52.444)\sigma_{R} \\ (R = 0.931, n = 9, P > 90\%)$$
(15)

$$\nu C = C_{op}^{(cm^{-1})} = 544.890(\pm 16.482) - 55.748(\pm 19.741)F - 32.598(\pm 11.923)R (R = 0.949, n = 9, P > 90\%)$$
(16)

<sup>1</sup>H NMR spectral study

The <sup>1</sup>H NMR spectra of synthesized chalcones were recorded in deuteriochloroform solutions employing tetramethylsilane (TMS) as internal standard. The signals of the ethylenic protons were assigned from their spectra. They were calculated as AB or AA' or BB' systems respectively. The lower chemical shifts (ppm) obtained for H<sub>α</sub> and higher chemical shifts (ppm) obtained for H<sub>β</sub> in this series of ketones. The vinyl protons give an AB pattern and the β-proton doublets were well separated from the signals of the aromatic protons. The assigned vinyl proton chemical shifts  $\delta$ (ppm) of all ketones were presented in Table 5.

In nuclear magnetic resonance spectra, the proton or the  ${}^{13}C$  chemical shifts ( $\delta$ ) depends on the electronic environment of the nuclei concerned. The assigned vinyl proton chemical shifts (ppm) have been correlated with reactivity parameters using Hammett equation in the form of

**Table 5** NMR chemical shifts δ(ppm) of substituted styryl 4-iodophenyl ketones (**entries 30– 40**).

Entry	Х	$\delta H_{lpha}$	$\delta H_{\beta}$	Х	$\delta C_{\alpha}$	$\delta C_{\beta}$	δCO	X
30	Н	7.740	7.877	-	121.48	145.47	189.75	-
31	4-Br	7.564	7.878	-	121.90	144.00	189.44	-
32	4-Cl	7.579	7.879	-	121.81	143.95	189.25	-
33	2-F	7.745	7.883	-	122.68	138.00	189.61	-
34	4-F	7.393	7.780	-	121.14	144.13	189.50	-
35	4-0H	7.668	7.837	4.867	121.81	143.95	189.25	-
36	2-	7.554	8.113	3.923	120.81	141.09	190.41	55.60
	OCH <sub>3</sub>							
37	4-	7.425	7.799	3.864	120.45	145.59	189.33	55.47
	OCH <sub>3</sub>							
38	$4-CH_3$	6.943	7.604	2.401	119.12	145.33	189.74	21.61

 $\log \delta = \log \delta_0 + \rho \sigma \tag{17}$ 

where  $\delta_0$  is the chemical shift of unsubstitued ketones.

The assigned H<sub> $\alpha$ </sub> and H<sub> $\beta$ </sub> proton chemical shifts (ppm) are correlated with various Hammett sigma constants. The results of statistical analysis [2,3,5,9,12,27–29,47–49] are presented in Table 6. The obtained correlations were poor for H<sub> $\alpha$ </sub> and H<sub> $\beta$ </sub> with Hammett  $\sigma$  constants, *F* and *R* parameters. The failure in correlation for both the proton chemical shifts and is due to the reasons stated in earlier and the conjugative structure shown in Fig. 4.

Application of Swain–Lupton's [53]treatment to the relative chemical shifts of  $H_{\alpha}$  and  $H_{\beta}$  with *F* and *R* values is successful with resonance, inductive and fail with *F* and *R* parameter generates the multiregression equations

$$\begin{split} \zeta_{H\alpha}^{(\text{ppm})} &= 7.375(\pm 0.177) + 1.459(\pm 0.511)\sigma_{I} \\ &+ 0.910(\pm 0.517)\sigma_{R} \\ &(R = 0.935, n = 9, P > 90\%) \end{split} \tag{18}$$

$$\begin{aligned} \delta^{(10000)}_{H\alpha} &= 7.416(\pm 0.183) + 0.331(\pm 0.044)F \\ &+ 1.060(\pm 0.670)R \\ &(R = 0.932, n = 9, P > 90\%) \end{aligned} \tag{19}$$

$$\begin{split} \delta^{(\text{ppm})}_{\text{H}\beta} &= 7.749(\pm 0.092) + 0.955(\pm 0.265)\sigma_{I} \\ &\quad -0.229(\pm 0.026)\sigma_{R} \\ &\quad (R = 0.946, n = 9, P > 90\%) \end{split} \tag{20}$$

$$\begin{aligned} {}^{(\text{pph})}_{\text{H}\beta} &= 7.801(\pm 0.102) + 0.049(\pm 0.002)F \\ &\quad - 0.078(\pm 0.002)R \\ &\quad (R = 0.922, n = 9, P > 90\%) \end{aligned} \tag{21}$$

<sup>13</sup>C NMR spectral study

Spectral analysts, organic chemists and scientists [2,3,5,9,12,27–29,47–50] have made extensive study of <sup>13</sup>C NMR spectra for a large number of different ketones and styrenes. The assigned vinyl  $C_{\alpha}$ ,  $C_{\beta}$  and carbonyl carbon chemical shifts are presented in Table 5. The results of statistical analysis are given in Table 6. The  $C_{\alpha}$  chemical shifts(ppm) gave satisfactory correlation with Hammett substituent constants  $\sigma(r = 0.945)$ ,  $\sigma^+(0.908)$ . The chemical shifts (ppm) of  $C_{\beta}$  carbon with Hammett  $\sigma$  constants, *F* and *R* parameters were gave poor correlation with negative  $\rho$  values. This is due to reasons stated earlier with the resonance conjugative structure shown in Fig. 4. The carbonyl carbon chemical shifts (ppm) of all ketones gave poor correlation with Hammett  $\sigma$  constants, *F* and *R* parameters.

#### Table 6

Results of statistical analysis of infrared  $\delta$ (ppm) of ethylenic protons, carbons and carbonyl carbons of substituted styryl 4-iodophenyl ketones (**entries 30–38**) with Hammett  $\sigma$ ,  $\sigma^+$ ,  $\sigma_L$ ,  $\sigma_R$  constants and *F* and *R* parameters.

Frequency	Constants	r	Ι	ρ	S	n	Correlated derivatives
$\delta^{(\text{ppm})}_{H}$	σ	0.813	7.517	0.131	0.26	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
110	$\sigma^{*}$	0.842	7.553	0.201	0.23	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_I$	0.834	7.373	0.454	0.24	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.824	7.438	-0.238	0.25	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.835	7.404	0.297	0.24	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.814	7.454	-0.144	0.26	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
$\delta^{(\mathrm{ppm})}_{\mathrm{Hs}}$	$\sigma$	0.798	7.846	-0.097	0.13	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
ч	$\sigma^{*}$	0.822	7.862	0.057	0.13	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_{I}$	0.825	7.784	0.216	0.13	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.844	7.762	-0.280	0.12	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.821	7.816	0.093	0.14	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.798	7.807	-0.108	0.13	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
$\delta^{(\text{ppm})}_{C_{\pi}}$	$\sigma$	0.945	121.31	1.849	0.98	7	H, 4-Br, 4-Cl, 2-F, 4-F, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub>
-4	$\sigma^{*}$	0.908	121.51	1.276	0.84	7	H, 4-Br, 4-Cl, 2-F, 4-F, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub>
	$\sigma_I$	0.864	120.28	3.125	0.84	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.812	121.95	-0.919	1.08	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.860	120.38	2.355	0.87	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.781	120.94	-0.742	1.08	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
$\delta^{(\mathrm{ppm})}_{C_{B}}$	$\sigma$	0.842	143.42	-1.952	2.58	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
h	$\sigma^*$	0.846	143.04	-2.206	2.32	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_{I}$	0.849	145.26	-5.737	2.28	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.857	145.59	6.686	2.16	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.859	145.51	-5.539	2.11	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.845	145.29	4.476	2.34	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
$\delta^{(\mathrm{ppm})}_{\mathrm{CO}}$	$\sigma$	0.833	189.56	-0.479	0.36	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma^{*}$	0.803	189.58	-0.027	0.38	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_I$	0.829	189.73	-0.491	0.37	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	$\sigma_R$	0.829	189.47	-0.362	0.37	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	F	0.825	189.71	-0.354	0.37	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>
	R	0.803	189.60	0.049	0.38	9	H, 4-Br, 4-Cl, 2-F, 4-F, 4-OH, 2-OCH <sub>3</sub> , 4-OCH <sub>3</sub> , 4-CH <sub>3</sub>

*r* = correlation coefficient;  $\rho$  = slope; *I* = intercept; *s* = standard deviation; *n* = number of substituents.

The Swain Lupton's [53] parameter correlations were satisfactorily obtained within these carbon chemical shifts and the regression equations are given in

 $\delta C_{\alpha}^{(\text{ppm})} = 120.449(\pm 0.596) + 3.693(\pm 1.723)\sigma_{I} + 1.078(\pm 0.745)\sigma_{R} \quad (R)$ = 0.967, n = 9, P > 95%)(22)

$$\begin{split} \delta C^{(\text{ppm})}_{\alpha} &= 120.605(\pm 20.623) + 2.958(\pm 1.503)F \\ &\quad + 1.086(\pm 0.585)R \quad (R \\ &= 0.964, n = 9, P > 95\%) \end{split} \tag{23}$$

$$\begin{split} \delta C^{(\text{ppm})}_{\beta} &= 146.021 (\pm 14.520) - 3.094 (\pm 0.390) \sigma_{I} \\ &+ 5.013 (\pm 1.447) \sigma_{R} \quad (R \\ &= 0.965, n = 9, P > 95\%) \end{split} \tag{24}$$

$$\delta C_{\beta}^{(\text{ppm})} = 145.833(\pm 11.539) - 4.648(\pm 1.711)F + 1.603(\pm 0.415)R \quad (R = 0.961, n = 9, P > 95\%)$$
(25)  
$$\delta CO_{\beta}^{(\text{ppm})} = 180.604(\pm 20.242) = 0.954(\pm 0.060)\sigma$$

$$\sigma CO^{(11)} = 189.004(\pm 20.242) - 0.954(\pm 0.069)\sigma_{I}$$
  
- 0.878(±0.070) $\sigma_{R}$  (R  
= 0.952, n = 9, P > 95%) (26)

$$\begin{split} \delta \text{CO}^{(\text{ppm})} &= 189.664(\pm 20.272) - 0.497(\pm 0.065) F \\ &\quad -1.258(\pm 0.603) R \quad (R \\ &\quad = 0.920, n = 9, P > 90\%) \end{split} \tag{27}$$

#### Antimicrobial activities

Chalcones possess a wide range of biological activities such as antibacterial [55], antifungal [55], antiviral [56], antifeedant [57], anticancer [55], antimalarial [58], antituberclosis [59], antiAIDS [60] and antioxidant [61] activities. These multipronged activities present in different chalcones are examined against respective microbes – bacteria's and fungi.

#### Antibacterial sensitivity assay

Antibacterial sensitivity assay was performed using Kirby–Bauer [62] disc diffusion technique. In each Petri plate about 0.5 mL of the test bacterial sample was spread uniformly over the Solidified Mueller Hinton agar using sterile glass spreader. Then the discs with 5 mm diameter made up of Whatmann No. 1 filter paper, impregnated with the solution of the compound (15 mg per 1 mL) were placed on the medium using sterile forceps. The plates were incubated for 24 h at 37 °C by keeping the plates upside down to prevent the collection of water droplets over the medium. After 24 h, the plates were visually examined and the diameter values of the zone of inhibition were measured. Triplicate results were recorded by repeating the same procedure.

The antibacterial screening effect of synthesized chalcones is shown in Fig. 6, Plates (1–10). The zone of inhibition is compared using Table 7. A good antibacterial activity has been possessed by all substituents on the microorganisms in general. All the compounds except those with 4-Cl and 4-OCH<sub>3</sub> substituents, showed good activities on *micrococcus luteus* species. All the compounds except those with H and 2-F substituents, showed good activities against *Escherichia coli*. The compounds with 4-F, 4-OH and 4-OCH<sub>3</sub> substituents have improved antibacterial activity against



PLATE-1





PLATE-3



PLATE-4



PLATE-5



PLATE-6



PLATE-7





PLATE-9 PLATE-10

Fig. 6. Antibacterial activity of substituted styryl 4-iodophenyl ketones-petri dishes.

 Table 7

 The antibacterial activities of styryl 4-iodophenyl ketones (entries 30–38).

Entry	Х	Zone of in	hibition (m	m)			
		Gram positive bacteria			Gram negative bacteria		
		B. subtilis	M. luteus	S. aureus	E. coli	P. aeruginosa	
30	Н	7	8	7	-	-	
31	4-Br	-	7	-	8	7	
32	4-Cl	8	-	7	8	7	
33	2-F	7	8	-	-	-	
34	4-F	8	7	8	7	8	
35	4-0H	12	10	7	7	8	
36	$2-OCH_3$	13	7	-	8	-	
37	$4-OCH_3$	12	-	7	7	8	
38	4-CH <sub>3</sub>	11	8	8	7	7	
	Ampicillin	19	17	13	16	14	
	DMSO	-	-	-	-	-	

*Pseudomonas aerogenosa.* The compounds with 4-F and  $4-CH_3$  substituents showed moderate activity against *Staphylococcus aureus.* All the compounds except those with 4-Br substituent, showed very good activities on *Bacillus subtillis* species.

# Antifungal sensitivity assay

Antifungal sensitivity assay was performed using Kirby–Bauer [62] disc diffusion technique. PDA medium was prepared and sterilized as above. It was poured (ear bearing heating condition) in the Petri-plate which was already filled with 1 mL of the fungal species. The plate was rotated clockwise and counter clock-wise for uniform spreading of the species. The discs were impregnated with the test solution. The test solution was prepared by dissolving 15 mg of the chalcone in 1 mL of DMSO solvent. The medium was allowed to solidify and kept for 24 h. Then the plates were visually examined and the diameter values of zone of inhibition were measured. Triplicate results were recorded by repeating the same procedure.

The antifungal activities of substituted chalcones synthesized in the present study are shown in Fig. 7 for Plates (1-4) and the zone of inhibition values of the effect is given in Table 8. The clustered

Table 8			
Antifungal activities of substituted	styryl 4-iodophenyl	ketones (entries	<b>30–38</b> ).

S. No.	Х	Zone of Inhibition (mm)				
		A. niger	Pen. Scup			
30	Н	6	11			
31	4-Br	-	9			
32	4-Cl	-	-			
33	2-F	8	-			
34	4-F	7	8			
35	4-0H	7	12			
36	2-OCH <sub>3</sub>	-	11			
37	4-OCH <sub>3</sub>	6	10			
38	4-CH <sub>3</sub>	-	-			
	Miconazole	12	13			
	DMSO	-	-			

column chart reveals that only a few compounds under investigation have excellent antifungal activity against all the two fungal species namely *Aspergillus niger*, and *pen.scup*. The Chalcones with —H, 4-Br, 4-F, 4-OH, 2-OCH<sub>3</sub> and 4-OCH<sub>3</sub> substituents have shown appreciable antifungal activity against *pen.scup*. Compound containing 2-F substituent alone has shown good activity against *A. niger*.

# Conclusions

We have developed an efficient crossed-aldol condensation for synthesis of chalcones using a versatile solid  $SiO_2-H_3PO_4$  acid catalyst. The yield of the reaction is more than 85%. The effects of substituent on the group frequencies and the antimicrobial activities of 4'-iodophenyl chalcones have been studied.

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PLATE-3

PLATE-4

Fig. 7. Antifungal activity of substituted styryl 4-iodophenyl ketones-petri dishes.

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