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RESEARCH ARTICLE

Synthesis of Biologically Active Chalcones of Substituted Indole-3 Carbaldehyde under Ultrasonic Irradiation

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ABSTRACT

A facile and ecofriendly synthesis of a series of novelindolylchalcones (3a–m) by the reaction of 3-formyl-2-phenylindoles with active methyl compounds 1, 3-cyclohexadione and dimedoneunder ultrasound irradiation. Application of ultrasound irradiation leads to many remarkable advantage, such as catalyst free reaction condition, shorter reaction time, simple work up procedure with advance 'green chemistry' approach. All the synthesized compounds have been characterized by spectral data analysis (IR, NMR, and MASS).

Key words: *Indole, 3-formyl-2-phenylindole, Ultrasound, Green chemistry*

INTRODUCTION

Indole nucleus is frequently found in medicinal chemistry and is considered as "privileged scaffolds". Perusal of literature revealed that the indole moiety is probably the most common and important feature of a variety of natural products and medicinal agents with significant biological activities including antioxidant, analgesic, antipyretic, antifungal, anthelmintic, cardiovascular, anticonvulsant, antimicrobial and selective COX-2 inhibitory activities. Hydroxyl indole plays a prominent role in the central nervous system and has been reported to product long lasting depletions of serotonin in brain tissues (Deschenes, *et al.,* 1999). Several mono and di hydroxy derivatives of indole have also been found to play an important role in brain as neurotransmitters. 2-aryl substituted indoles have been implicated in inhibition of bacterial histidine kinases (Gaikwad, *et al.,* 2010). Another example of an active compound of this class would be 3-phenylindole which is an inhibitor of brassinin glucosyltransferase, a phytoalexin detoxifying enzyme from the fungus, Sclerotinia sclerotiorum (Joshi, *et al.,* 1978). Indole-based derivatives have been investigated for anticancer activities. Indole-3-carbinols have been previously reported to exhibit anticancer activities against a number of human cancers through acting on different cellular signaling pathways (Kaufmann, *et al.,* 2007). 1-Aroylindoles and 3-aroylindoles have shown potent cytotoxicity against different human cancer cell lines (Kaufmann, *et al.,* 2007).

Chalcones represent an important group of natural compounds with a variety of biological activities including antibacterial, antimalarial, antioxidants and antifungal ones. They have found numerous applications as pesticides, photoprotectors in plastics, solar creams, food additives as well as anti- inflammatory and anticancer agents. Moreover, the chalcone framework could be easily incorporated into the more complex structures in order to design new potentially bioactive compounds (Gaikwad, *et al.*, 2010).

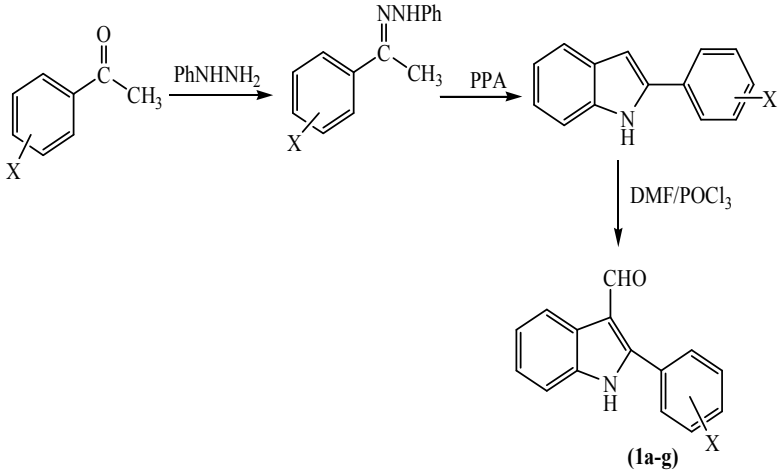
Therefore, the synthesis and selective functionalization of indoles have been the focus of active research over the years. The indole derivatives have been widely studied, α, β-unsaturated ketones of chalcone types containing this heterocycle in which products of crotonic condensation of 3-formyl indole derivatives with different acetophenones were described previously in basic media using mostly piperidine as a catalyst. These compounds are usually synthesized by the Claisen–Schmidt condensation of aromatic aldehydes with methyl ketones in the presence of bases such as KOH, LiHDMS and calcined $NaNO_3$/ natural phosphates. The acid

catalyzed methodologies include the use of Zeolites, K_3PO_4 and BF_3–Et_2O (Narender, and Reddy 2007).

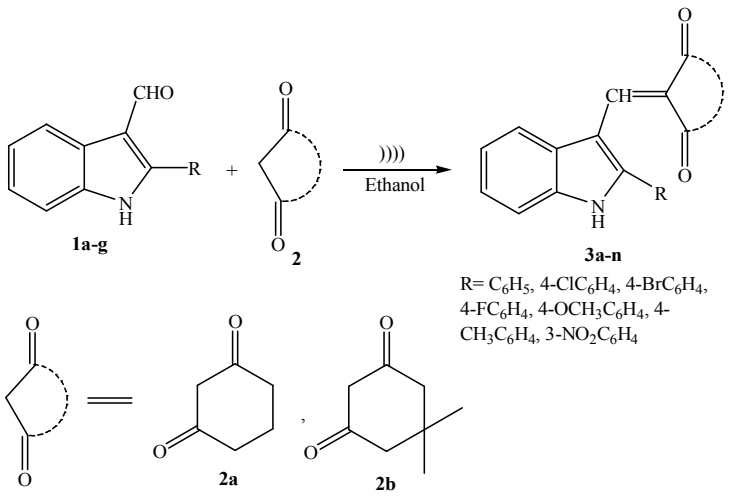
In recent years, ultrasound has become a highly useful method for performing a wide range of chemical reactions and processes, including chemical synthesis, materials production and water treatment. Ultrasound works by the phenomenon of cavitation; which involves the growth, oscillation, and collapse of bubbles under the action of an acoustic field. The collapse causes a couple of strong physical effects outside the bubble such as-shear forces, jets and shock waves. These cavitation-induced effects can cause physical, chemical, and biological transformations more effectively. Thus, ultrasound has found applications in chemistry, in materials, in life sciences as well as in medicine (Mason, 2007).

Hence, in continuation of our interest on indole derivatives, we have chosen these molecular entities to suitably combine them through carbon–carbon bond formation for creating new hybrid molecules (Pathak, *et al.,* 2001). A series of chalcone derivatives were synthesized by one-pot reaction of 3-formyl-2-phenylindole (1a-g), 1,3-cyclohexanedione (2a), dimedone(2b) and base (NaOH) in ethanol under ultrasound irradiation.

Scheme 1



Scheme 2



RESULT AND DISCUSSION

2-Phenylindole derivatives have been synthesized by the method of Joshi, *et al*., 1978. To achieve suitable conditions for the synthesis of chalcones of indolaldehyde, we choose 3-formyl-2-phenylindole (1a) and 1, 3-cyclohexadione (2a) as a model reaction in different solvents such as methanol, ethanol, acetonitrile, and toluene but we found that ethanol was a suitable solvent for our reaction.

Further we explore our reaction by using different derivatives of 3-formyl-2-phenylindole (1ag) and 1,3-cyclohexadione (2a), (Table1) and 5,5-dimethyl-1,3-cyclohexadione(2b), (Table 2) for the synthesis of chalcone derivatives (3a-3n).

The results in table1 and 2 show that a number of derivatives of 3-formyl-2-phenylindole including electron withdrawing and electron-donating group and active methylene compounds dimedone and 1, 3-cyclohexadione were allowed to participate in the reaction to form chalcones under ultrasound irradiation.

Table 1: Result and condition of the synthesis of compounds 3a-g by the reaction of 3-formyl-2-phenylindole and 1, 3- cyclohexadione

S.No.	Compound	R	Time (min)		M.P. (^{0}C)	Yield (%)*		Mol. Formula
			Con.	U I		Con.	U I	
1	3a	C_6H_5	57	19	189-191	72	76	$C_{21}H_{17}NO_2$
2	3b	4-ClC_6H_4	54	18	274-276	65	78	$C_{21}H_{16}NClO_2$
3	3c	4-FC_6H_4	47	15	280-282	62	81	$C_{21}H_{16}NFO_2$
4	3d	4-BrC_6H_4	42	20	286-288	71	79	$C_{21}H_{16}NBrO_2$
5	3e	4-$OCH_3C_6H_4$	40	16	242-244	62	77	$C_{22}H_{19}NO_3$
6	3f	4-$CH_3C_6H_4$	57	17	258-260	68	76	$C_{22}H_{19}NO_2$
7	3g	3-$NO_2C_6H_4$	45	16	310-312	65	80	$C_{21}H_{16}N_2O_4$

* Isolated yield, Con.:– Conventional, U I:- Ultrasound Irradiation

Table 2: Synthesis of compounds 3h-n by the reaction of 3-formyl-2-phenylindole and 5,5-dimethyl-1,3-cyclohexadione

S.No.	Compound	R	Time (min)		M.P. (^{0}C)	Yield (%)*		Mol. Formula
			Con.	U I		Con.	U I	
1.	3h	C_6H_5	58	15	165-168	73	80	$C_{23}H_{21}NO_2$
2.	3i	4-ClC_6H_4	55	16	292-294	68	84	$C_{23}H_{20}NClO_2$
3.	3j	4-FC_6H_4	45	13	>340	67	88	$C_{23}H_{20}NFO_2$
4.	3k	4-BrC_6H_4	48	12	300-302	70	86	$C_{23}H_{20}NBrO_2$
5.	3l	4-$OCH_3C_6H_4$	42	16	308-310	65	83	$C_{24}H_{23}NO_3$
6.	3m	4-$CH_3C_6H_4$	56	12	284-286	68	81	$C_{24}H_{23}NO_2$
7.	3n	3-$NO_2C_6H_4$	46	14	>340	63	87	$C_{23}H_{20}N_2O_4$

* Isolated yield, Con.:– Conventional, U I: - ultrasound irradiation

EXPERIMENTAL

The purity of the compounds was checked by TLC using silica gel-G as adsorbent, UV light or Iodine accomplished visualization. IR spectra were recorded on a Shimadzu FT IR- 8400S spectrophotometer using KBr pellets. ^{1}H NMR spectra were recorded in DMSO-d_6 using TMS as an internal standard on a Bruker spectrophotometer at 300 MHz. Mass spectrum of representative compound was recorded on Waters Xevo Q-Tof spectrometer at 70 eV. Ultrasound irradiation reaction was carried out in ultrasonic bath (Toshniwal-sw-4) operating at 150 w generating 37 KHz output frequency. All the Melting points were determined using an open-ended capillary tube method and are uncorrected.

1. General Procedure for the Synthesis of 3-Formyl-2-Phenylindole(1a-g):

Synthesis of 3-formyl-2-(substitutedphenyl)-indole (1a-g) was carried out by Vielsmayer Haack reaction. Phosphorus oxychloride 0.8mL (8.7mmol) was mixed with 3.5 mL of ice-cold dimethylformamide. To this mixture, 1.0 g (6.8 mmol) of 2-phenylindole derivatives dissolved in 23 mL of ice-cold dimethylformamide was added under magnetic stirring. The compounds reacted for 1 h at 40 ^{0}C. To the pale yellow reaction mixture, 35 mL of ice-cold water was added, and the color changed to burgundy. After basification with 5% aqueous sodium hydroxide, the color of the solution again changed to yellow, and an additional 30 mL of water was added. Thus obtained, resultant was heated on steam bath for 1 hr. It was cooled, filtered and crystalised from acetone.

Spectral Analysis of 3-Formyl- 2-Phenyl-1*H*-Indole (1a):
$C_{15}H_{11}NO$, Mol. Wt 221.25**;** IR (KBr)ν_{max} in cm^{-1} 3221, 3059, 1710, 1581; ^1H NMR (DMSO-d_6) δ in ppm 11.48 (s, 1H, NH), 9.96 (s, 1H, CHO), 7.21-7.80 (m, 9H, Ar-H); 222.25 $(M+H)^+$; Elemental Analysis Cacld. For $C_{15}H_{11}NO$, C 81.43, O 7.23, N 6.33, H 5.01; Found, C 81.40, O 7.22, N 6.35, H 5.03.

GENERAL METHOD (I):
(Ultrasound Irradiation) for Synthesis of 2-(2-Phenyl-1*H*- indol-3- ylmethylene)-cyclohexane-1, 3-dione derivatives (3a-n):
A mixture of 3-formyl-2-phenylindole (1a-i), 1,3-cyclohexadione (2a) (3 mmol) or 5,5-dimethyl-1,3-cyclohexadione (2b) (3 mmol), base (NaOH) and ethanol (15ml) was taken in a conical flask and sonicate for appropriate time given in table (2). After completion of the reaction (checked by TLC), thus obtained resultant mixture was filtered and crystallized by ethanol.

METHOD (II):
(Conventional Procedure) for Synthesis of 2-(2-Phenyl-1*H*-indol-3-ylmethylene)-cyclohexane-1, 3-dione derivatives (3a-n):
2-Phenyl-1*H*-indole-3-carbaldehyde (1a-i) (3 mmol) and 2a or 2b (3 mmol) were dissolved in minimum amount of ethanol (25 ml) and then stirred at room temperature for 5 min. Sufficient 2N NaOH solution (40 ml) was added to it and whole reaction mixture was further stirred for half an hour, neutralized with 2N HCl (3ml) diluted with water and left overnight to get solid compound which was filtered and recrystallized from ethanol to obtain pure compound tabulated in (Table-2).

SPECTRAL DATA:
2-(2-phenyl-1*H*-indol-3-ylmethylene)-cyclohexane-1,3-dione (3a):
$C_{21}H_{17}NO_2$, Mol. Wt 315.37 IR (KBr)ν_{max} in cm^{-1} 3180, 3060, 1685, 1550 ; ^1H NMR(DMSO-d_6) δ in ppm 11.34 (s, 1H, NH), 8.35 (s, 1H, C=C), 7.10-7.35 (m, 9H, Ar-H), 1.65-2.43 (m, 6H, 3xCH_2); 316.37 $(M+H)^+$; Elemental Analysis Cacld. for $C_{21}H_{17}NO_2$, C 79.98, O 10.15, H 5.43, N 4.44; Found, C 79.95, O 10.16, H 5.47, N 4.42.

2-[2-(4-Chloro-phenyl)-1*H*-indol-3-ylmethylene]-cyclohexane-1,3-dione(3b):
$C_{21}H_{16}NClO_2$, Mol. Wt 349.81, IR (KBr)ν_{max} in cm^{-1} 3340, 3050, 1680, 1555, 670; ^1H NMR(DMSO-d_6) δ in ppm 11.34 (s, 1H, NH), 8.28 (s, 1H, C=C), 7.00-7.48 (m, 8H, Ar-H), 1.73-2.45 (m, 6H, 3xCH_2); 350.81 $(M+H)^+$. Elemental analysis Cacld. For $C_{21}H_{16}NClO_2$, C 72.10, Cl 10.13, O 9.15, H 4.13, N 4.00; Found, C 72.12, Cl 10.11, O 9.16, H 4.63, N 3.98

2-[2-(4-Fluoro-phenyl)-1*H*-indol-3-ylmethylene]-cyclohexane-1,3-dione (3c):
$C_{21}H_{16}NFO_2$, Mol. Wt 333.36, IR (KBr)ν_{max} in cm^{-1} 3340, 3045, 1690, 1560, 1045; ^1H NMR(DMSO-d_6) δ in ppm 10.65 (s, 1H, NH), 8.38(s, 1H, C=C), 7.30-7.64 (m, 8H, Ar-H), 1.70-2.51 (m, 6H, 3xCH_2); 334.36 $(M+H)^+$. Elemental analysis Cacld For $C_{21}H_{16}NFO_2$, C 75.66, O 9.60, F 5.70, H 4.84, N 4.20; Found, C 75.63, O 9.59, F 5.73 H 4.83, N 4.22

2-[2-(4-Bromo-phenyl)-1*H*-indol-3-ylmethylene]-cyclohexane-1,3-dione (3d):
$C_{21}H_{16}NBrO_2$, Mol. Wt 393.26, IR (KBr)ν_{max} in cm^{-1} 3350, 3055, 1690, 1545, 550(C-Br) ; ^1H NMR(DMSO-d_6) δ in ppm 10.85 (s, 1H, NH), 8.40(s, 1H, C=C), 7.30-7.95 (m, 8H, Ar-H), 1.60-2.38 (m, 6H, 3xCH_2) ; 394.26 $(M+H)^+$. Elemental analysis Cacld For $C_{21}H_{16}NBrO_2$, C 63.97, Br 20.27, O 8.12, H 4.09, N 3.55; Found, C 63.99, Br 20.28, O 8.10, H 4.07, N 3.56

2-[2-(4-Methoxy-phenyl)-1*H*-indol-3-ylmethylene]-cyclohexane-1,3-dione(3e):
$C_{22}H_{19}NO_3$, Mol. Wt 345.39, IR (KBr)ν_{max} in cm^{-1} 3350, 3045, 1695, 1550, 1110 ; ^1H NMR(DMSO-d_6) δ in ppm 10.85 (s, 1H, NH), 8.40(s, 1H, C=C), 6.80-7.40 (m, 8H, Ar-H), 3.70(s, 3H, CH_3), 1.85-2.59 (m, 6H, 3xCH_2) ; 345.39 $(M+H)^+$; Elemental analysis Cacld For $C_{22}H_{19}NO_3$, C 76.50, O 13.90, H 5.54, N 4.06 ; Found, C 76.53, O 13.88, H 5.55, N 4.08.

2-[2-(4-Methyl-phenyl)-1*H*-indol-3-ylmethylene]-cyclohexane-1,3-dione (3f):
$C_{22}H_{19}NO_2$, Mol. Wt 329.39, IR (KBr)ν_{max} in cm^{-1} 3270, 3045, 1690, 1565 ; ^1H NMR(DMSO-d_6) δ in ppm 10.95 (s, 1H, NH), 8.40(s, 1H, C=C), 7.10-7.50 (m, 8H, Ar-H), 2.38(s, 3H, CH_3) 1.76-2.22 (m,

6H, 3xCH_2) ; 330.39 $(M+H)^+$; Elemental analysis Cacld For $C_{22}H_{19}NO_2$, C 80.22, O 9.71, H 5.81, N 4.25 ; Found, C 80.25, O 9.69, H 5.79, N 4.27

2-[2-(3-Nitro-phenyl)-1*H*-indol-3-ylmethylene]-cyclohexane-1,3-dione (3g):
$C_{21}H_{16}N_2O_4$, Mol. Wt 360.36, IR (KBr)ν_{max} in cm^{-1} 3355, 3085, 1680, 1610, 1540 ; ^{1}H NMR(DMSO-d_6) δ in ppm 11.43 (s, 1H, NH), 8.40 (s, 1H, C=C), 6.85-8.10 (m,8 H, Ar-H), 1.76-2.40 (m, 6H, 3xCH_2) ; 361.36 $(M+H)^+$. Elemental analysis Calcd For $C_{21}H_{16}N_2O_4$, C 69.99, O 17.76, N 7.77, H 4.48 ; Found, C 69.95, O 17.77, N 7.79, H 4.49

5,5-Dimethyl-2-(2-phenyl-1*H*-indol-3-ylmethylene)-cyclohexane-1,3-dione(3h):
$C_{23}H_{21}NO_2$, Mol. Wt 343.42, IR (KBr)ν_{max} in cm^{-1} 3345, 3060, 1690, 1535 ; ^{1}H NMR(DMSO-d_6) δ in ppm 11.43 (s, 1H, NH), 8.35 (s, 1H, C=C), 7.10-7.45 (m, 9H, Ar-H), 2.02-2.59 (m, 4H, 2xCH_2), 0.97 (s, 6H, CH_3) ; 344.42 $(M+H)^+$. Elemental analysis Cacld For $C_{23}H_{21}NO_2$, C 80.44, O 9.32, H 6.16, N 4.08; Found, C 80.46, O 9.35, H 6.13, N 4.06

2-[2-(4-Chloro-phenyl)-1*H*-indol-3-ylmethylene]-5,5-dimethylcyclohexane-1,3-dione(3i):
$C_{23}H_{20}NClO_2$, Mol. Wt 377.86, IR (KBr)ν_{max} in cm^{-1} 3340, 3050, 1680, 1545, 675 ; ^{1}H NMR(DMSO-d_6) δ in ppm 11.77 (s, 1H, NH), 8.38 (s, 1H, C=C), 7.00-7.65 (m, 8H, Ar-H), 2.10-2.45 (m, 4H, 2xCH_2), 1.10 (s, 6H, CH_3) ;378.86 $(M+H)^+$; Elemental analysis Cacld For $C_{23}H_{20}NClO_2$, C 73.11, Cl 9.38, O 8.47, H 5.33, N 3.77; Found, C 73.08, Cl 9.39, O 8.45, H 5.37, N 3.77.

2-[2-(4-Fluoro-phenyl)-1*H*-indol-3-ylmethylene]-5,5-dimethylcyclohexane-1,3-dione(3j):
$C_{23}H_{20}NFO_2$, Mol. Wt 361.41, IR (KBr)ν_{max} in cm^{-1} 3360, 3045, 1685, 1545, 1080 ; ^{1}H NMR(DMSO-d_6) δ in ppm 11.43 (s, 1H, NH), 8.40 (s, 1H, C=C), 7.05-7.58 (m, 8H, Ar-H), 2.05-2.39 (m, 4H, 2xCH_2), 1.04 (s, 6H, CH_3); 362.41 $(M+H)^+$; Elemental analysisCacld For $C_{23}H_{20}NFO_2$, C 76.44, O 8.85, H 5.58, F 5.26, N 3.88 ; Found, C 76.46, O 8.88, H 5.55, F 5.26, N 3.85.

2-[2-(4-Bromo-phenyl)-1*H*-indol-3-ylmethylene]-5,5-dimethylcyclohexane-1,3dione(3k):
$C_{23}H_{20}NBrO_2$, Mol. Wt 422.31, IR (KBr)ν_{max} in cm^{-1} 3365, 3040, 1695, 1535, 580 ; ^{1}H NMR(DMSO-d_6) δ in ppm 10.95 (s, 1H, NH), 8.40 (s, 1H, C=C), 7.28-7.76 (m, 8H, Ar-H), 2.25-2.48 (m, 4H, 2xCH_2), 1.16 (s, 6H, CH_3) ; 423.31 $(M+H)^+$; Elemental analysisCacld For $C_{23}H_{20}NBrO_2$, C 65.41, Br 18.92, O 7.58, H 4.77, N 3.32 ; Found, C 65.43, Br 18.90, O 7.55, H 4.79, N 3.33.

2-[2-(4-Methoxy-phenyl)-1*H*-indol-3-ylmethylene]-5,5-dimethylcyclohexane-1,3-dione(3l):
$C_{24}H_{23}NO_3$, Mol. Wt 373.44, IR (KBr)ν_{max} in cm^{-1} 3340, 3050, , 1680, 1525, 1230 ; ^{1}H NMR(DMSO-d_6) δ in ppm 11.05 (s, 1H, NH), 8.32 (s, 1H, C=C), 6.82-7.45 (m, 8H, Ar-H), 3.95 (s, 3H, OCH_3), 1.98-2.37 (m, 4H, 2xCH_2), 0.97 (s, 6H, CH_3) ; 374.44 $(M+H)^+$; Elemental analysis Cacld. For $C_{24}H_{23}NO_3$, C 77.19, O 12.85, H 6.21, N 3.75; Found, C 77.17, O 12.82, H 6.23, N 3.78.

2-[2-(4-Methly-phenyl)-1*H*-indol-3-ylmethylene]-5,5-dimethylcyclohexane-1,3-dione(3m):
$C_{24}H_{23}NO_2$, Mol. Wt 357.44, IR (KBr)ν_{max} in cm^{-1} 3355, 3035, 1685, 1520 ; ^{1}H NMR(DMSO-d_6) δ in ppm 10.88 (s, 1H, NH), 8.32 (s, 1H, C=C), 6.97-7.52 (m, 8H, Ar-H), 2.40-2.67 (m, 4H, 2xCH_2), 2.32 (s, 3H, CH_3), 1.16 (s, 6H, CH_3) ; 358.44 $(M+H)^+$; Elemental analysis Cacld. For $C_{24}H_{23}NO_2$, C 80.64, O 8.95, H 6.49, N 3.92; Found, C 80.66, O 8.96, H 6.45, N 3.93.

2-[2-(3-Nitro-phenyl)-1*H*-indol-3-ylmethylene]-5,5-dimethylcyclohexane-1,3-dione(3n):
$C_{23}H_{20}N_2O_4$, Mol. Wt 388.42, IR (KBr)ν_{max} in cm^{-1} 3350, 3060, 1695, 1540, 1260 ; ^{1}H NMR(DMSO-d_6) δ in ppm 11.25 (s, 1H, NH), 8.30 (s, 1H, C=C), 7.10-8.52 (m, 8H, Ar-H), 2.42-2.83 (m, 4H, 2xCH_2), 1.11 (s, 6H, CH_3) ; 389.42 $(M+H)^+$; Elemental analysis Cacld. For $C_{23}H_{20}N_2O_4$, C 71.12, O 16.48, N 7.21, H 5.19 ; Found, C 71.15, O 16.46, N 7.23, H 5.16.

CONCLUSION

We have synthesized chalcone derivatives by using 3-formyl-2-phenylindole, dimedone and 1,3-cyclohexadione in a small amount of 95% ethanol by employing sonication. We developed green procedure for the synthesis of chalcone derivatives under ultrasound irradiation. In addition, high yields of the products, short reaction times, ease of work-up, and low-cost make the above method advantageous in comparison to the traditional conventional method.

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