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### RESEARCH ARTICLE

## SYNTHESIS, CHARACTERIZATION AND STUDY OF BIOLOGICAL ACTIVITIES SOME NEW CHALCONE DERIVATIVES CONTAINING DIPHENYL AMINE MOIETY USING RECYCLABLE CATALYST SYSTEM.

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#### Abstract

In an effort to develop a series of substituted chalcone derivatives have been synthesized by Claisen – Schmidt condensation. In the present study some new chalcones have been synthesized by the reaction of acetyl diphenyl amine with different aromatic aldehydes in the presence of aqueous solution of sodium hydroxides ethanol and PEG 400 used as a catalyst at room temperature. The structure of various synthesized compounds are assigned on the basis of elemental analysis, IR, NMR and Mass spectral data, the synthesized compounds were screened for antimicrobial activity.

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#### Introduction:-

Heterocyclic compounds are widely distributed in nature and occupy a prominent place in medicinal, pharmaceuticals and as drug intermediate.

Chalcones structurally represent open chain flavonoid in which the two aromatic rings are joined by a three – carbon  $\alpha$ ,  $\beta$  - unsaturated carbonyl system (1, 3-diphenyl – 2 – propen – 1- one)

Chalcones possess different types of biological activity<sup>1</sup> such as antimicrobial<sup>2</sup> anti-inflammatory<sup>3</sup>, analgesic<sup>4</sup>, antiplatelet<sup>5</sup>, antiulcerative<sup>6</sup>, antifungal<sup>7</sup>, anti-malarial<sup>8</sup>, anti plasmodial, anti cancer<sup>9</sup> anti viral<sup>10</sup>, antileishmanial<sup>11</sup>, antioxidant, anti-tubercular activities<sup>12-15</sup>.

Chalcones and its derivatives are primarily synthesized in the laboratory using Claisen-Schmidt reaction, in which acetophenone or its derivatives is reacted with benzaldehyde or its derivatives using strong base. Such as NaOH or KOH or these days PEG is used as a catalyst, shown in following reaction.

#### Materials and methods:-

Acetyl-diphenyl amine and all aromatic aldehyde were purchased from Sigma Aldrich. PEG (400) used was of Thomas Backer. Ethanol and other chemicals of AR grade were used as received.

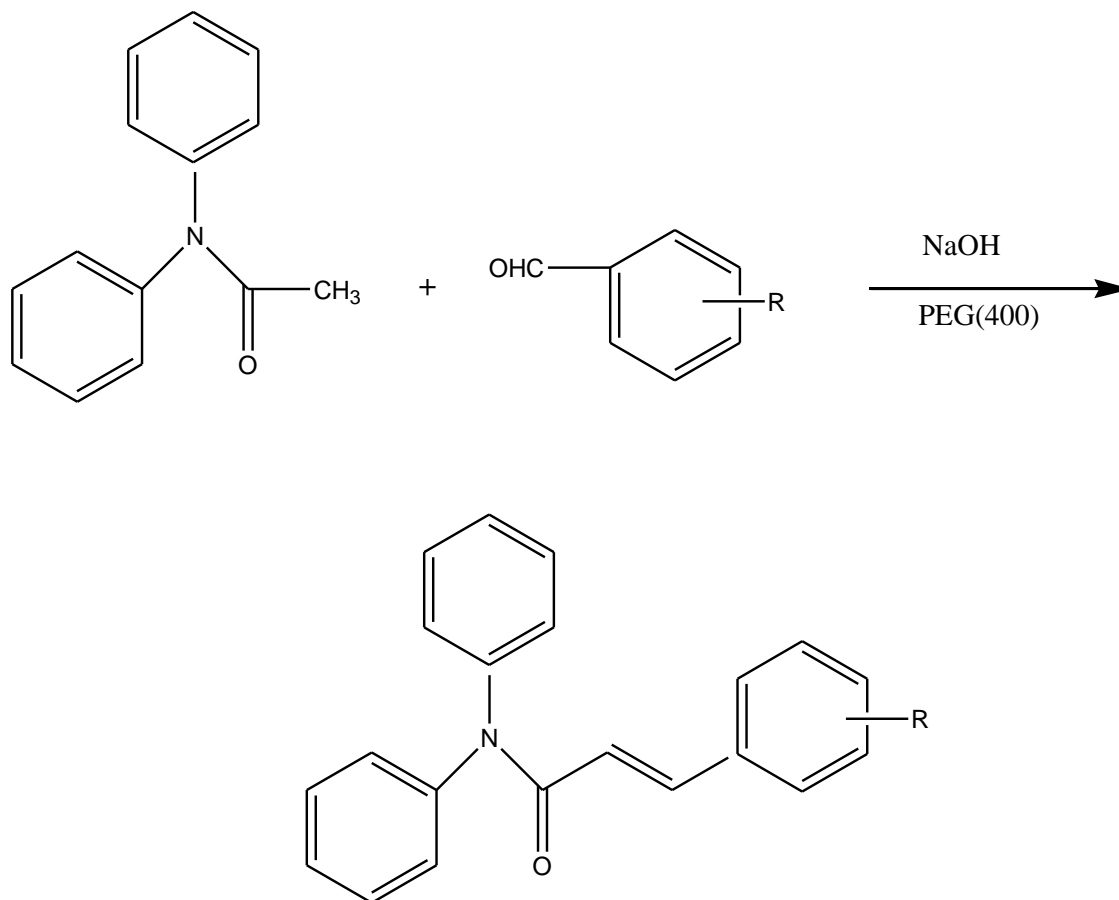
#### Experimental:-

Synthesis of chalcone is a single step method. A mixture of acetyl-diphenyl amine (0.01) (mol) and appropriate aldehyde (R) or 0.01 (mol) was stirred in ethanol and then aqueous solution of NaOH 40%, 10ml was added to it. PEG (400) was used as a catalyst. The reaction mixture was stirred for 2-3 hours.

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The sodium salt of chalcone separated was decomposed by ice-cold HCl (30%) chalcone was filtered, washed with water (50 ml) dried and recrystallized from absolute ethanol.



Where R = Substituted Aldehyde  
(Scheme – 1 Synthesis of some new chalcones of acetyl diphenyl amine)

The yield of the synthesized compounds was found to be significant in the presence of PEG(400) catalyst. Elemental analysis confirmed percentage of carbon, hydrogen and oxygen to be similar to the calculated values.

#### Physical measurements and analytical data:-

The melting point were determined in open capillaires on electro thermal apparatus and are uncorrected the purity of all compounds were checked by thin layer chromatography using protected silica 60F-254 plate using hexane, ethylacetate, chloroform as mobile phase.

**Table:-** Chalcone Obtained by the condensation of N-acetyl diphenylamine with various substituted aldehydes

S. No.	Substituted (R)	Molecular Formula	M.P. °C	% Yield	Carbon (%)		Hydrogen (%)		Nitrogen (%)		Oxygen (%)	
					Found	Calcd.	Found	Calcd.	Found	Calcd.	Found	Calcd.
1.	Benzaldehyde	C <sub>21</sub> H <sub>17</sub> NO	209	90	84.28	84.25	5.68	5.72	4.68	4.67	5.35	5.34
2.	4-methoxy Benz.	C <sub>22</sub> H <sub>19</sub> NO <sub>2</sub>	255	84	80.24	80.22	5.77	5.81	4.44	4.25	9.72	9.71
3.	3-bromo Benz.	C <sub>21</sub> H <sub>16</sub> BrNO	281	85	66.66	66.68	4.23	4.26	3.70	3.70	4.23	4.23
4.	3-methoxy 4-hyd Benz.	C <sub>22</sub> H <sub>19</sub> NO <sub>3</sub>	367	80	76.52	76.50	5.50	5.54	4.05	4.06	13.91	13.90
5.	2-chloro 6-fluro Benz.	C <sub>21</sub> H <sub>15</sub> ClFNO	265	81	71.79	71.70	4.27	4.30	3.98	3.98	4.55	4.55

Microanalysis of carbon, hydrogen and oxygen of the compounds were carried on a Heraeus Carlo Erba 1108 elemental analyzer. IR spectra were recorded on perkin-elemer RX-1 infrared spectrometer in the range 4000-450  $\text{cm}^{-1}$ ,  $^1\text{H}$ NMR,  $^{13}\text{C}$ -NMR spectra of the compounds were recorded on a Bruker Advance 300 MHz. The  $\text{ESI}^+$  mass spectra were recorded on a JEOL-Accu TOF JMS – 100LC mass spectrometer.

### Result and Discussion:-

That data obtained of the synthesized compounds.

**(diphenylamine-yl)-3-(phenyl)-prop-2 ene-1one-(a)** shiny yellow crystal, yield-90%, mp. 209°C, Anal. Caltd for  $\text{C}_{21}\text{H}_{17}\text{NO}$ , C=84.25, H=5.73 W=4.68, O=5.34(%) found C=84.28, H=5.68, N=4.68, O=5.35(%) IR ( $\text{cm}^{-1}$ ) 3423 (C=O) 1657 (C=C), 1402(C-O-C),  $^1\text{H}$ NMR (400MHz  $\text{CdCl}_2$ )  $\square$  (inppm), 7.85 (d;2H, Ar-H), 7.38-7.35(d, 2H, Ar-H), 7.25 (d, 1HRCH) 7.15 – 7.06 (m, 2H, S-H),  $^{13}\text{C}$ NMR (100 MHz;  $\text{CdCl}_2$ )  $\square$  (inppm) 168.89, 135.65, 134.01, 129.91, 129.53, 120.41-41, MS m/z 299( $\text{m}^+$ ).

**(diphenylamine-yl)-3-(4-methoxyphenyl)prop-2-ene-1-one (b)** light shiny brown, yield 84%, mp 225°C, and caltd for  $\text{C}_{22}\text{H}_{19}\text{NO}_2$  C=80.22, H=5.81, N=4.25, O=9.71 (%) found C=80.24, H=5.77, N=4.44, O=9.72(%) IR ( $\text{cm}^{-1}$ ) 1692 (C=O), 1562 (C=C) 1272 (C-O-C),  $^1\text{H}$ NMR (400 MH- $\text{CdCl}_2$ )  $\square$  (inppm) = 7.89, (S, 1H, CH), 7.42-7.37 (d2H Ar-H), 7.30 (S, 1H, CH), 7.20-1.13 (M, 2H, Ar-H),  $^{13}\text{C}$ -NMR (100 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) 172.40, 138.70, 135.02, 132.10, 130.80, 123.20 MS M/z 329 ( $\text{M}^+$ )

**(diphenyl amine-yl)-3-(3-bromo) Prop-2ene-1 one (c)** : light yellow, yield, 85%, M.P. – 281°C And Caltd for  $\text{C}_{21}\text{H}_{16}\text{BrNO}$ , C=66.68, H=4.23, Br=21.13, N=3.70, O=4.23(%) IR ( $\text{cm}^{-1}$ ), 1695 (C=O). 1565 (C=C), 1275 (C-O-C)  $^1\text{H}$ NMR (400 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) = 7.90 (S, 1H, CH), 7.45-7.38 (d, 2H, Ar-H) 7.32 (S, 1H, CH) 7.22-7.15 (M, 2H, Ar-H),  $^{13}\text{C}$ -NMR (100 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) = 190.90, 145.60, 140.02, 135.20, 132.40, 125.10, MS M/z 378 ( $\text{M}^+$ ).

**(diphenyl amine-yl)-3-(3-methoxy-4 Hydroxy Phenyl) Prop-2ene-1 one (d)** : light cream, yield, 80%, M.P. – 267°C Anal Caltd for  $\text{C}_{22}\text{H}_{19}\text{NO}_3$ , C=76.50, H=5.54, N=4.06, O=13.90(%) found – C=76.52, H=5.50, N=4.05, O=13.91% IR ( $\text{cm}^{-1}$ ), 1700 (C=O). 1575 (C=C), 1280 (C-O-C)  $^1\text{H}$ NMR (400 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) = 7.92 (S, 1H, CH), 7.50-7.42 (d, 2H, Ar-H) 7.35 (S, 1H, CH) 7.32-7.19 (M, 2H, Ar-H),  $^{13}\text{C}$ -NMR (100 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) = 175.80, 140.80, 137.10, 133.20, 132.10, 125.10, MS M/z 345 ( $\text{M}^+$ ).

**(diphenyl amine-yl)-3-(2-chloro-6-fluro) Prop-2ene-1 one (e)** : white, yield, 81%, M.P. – 265°C Anal Caltd for  $\text{C}_{21}\text{H}_{15}\text{ClFNO}$ , C=71.70, H=4.30, Cl=10.08, F=5.40, N=3.98, O=4.55, found C=71.79, H=4.27, Cl=10.11, F=5.41, N=3.98, O=4.55(%) IR ( $\text{cm}^{-1}$ ), 1720 (C=O). 1590 (C=C), 1295 (C-O-C);  $^1\text{H}$ NMR (400 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) = 7.98 (S, 1H, CH), 7.55-7.45 (d, 2H, Ar-H) 7.37 (S, 1H, CH) 7.35-7.25 (M, 2H, Ar-H),  $^{13}\text{C}$ -NMR (100 MHz  $\text{CdCl}_2$ )  $\square$  (inppm) = 177.90, 142.70, 138.20, 134.1, 133.20, 127.20 MS M/z 351 ( $\text{M}^+$ ).

### Biological Activity:-

The biological activity was performed by filter paper disc plate method.

### Antibacterial activity:-

All human pathogenic bacteria viz Klebsiella ( $\text{gm}^{+ve}$ ), Escherichia coli, staphylococci ( $\text{gm}^{+ve}$ )<sup>16</sup> were purchased from Ajay Diagnostic and Research centre Jay Hospital, Agra India.

### Antifungal Activity:-

The synthesized compounds were screened for their in vitro antifungal activity against Aspergillus niger, Aspergillus fumigatus, Candida albicans by measuring the zone of inhibition in mm.<sup>17-18</sup>

**Antibacterial & Antifungal data of compounds**Zone of inhibition measured in mm MIC = 10  $\mu$ g/disc.

Comp.	Antifacterial			Antifungal		
	Klebsiella pneumoniae	Escherichia coli	Staphylococcus aureus	Aspergillus niger	Asperigillus fumigatus	Candida albicans
a	08	07	08	08	07	07
b	07	06	06	07	08	06
c	06	07	08	06	07	08
d	08	06	09	08	09	07
e	08	07	08	06	08	06

**Application:-**

This is an environmentally benign procedure and reduces the total reaction time and gave excellent yield of chalcones.

**Conclusion:-**

The present work demonstrated the synthesis of chalcone in excellent yield in the presence of a reusable and environmentally benign reaction catalyst PEG(400), the structure of the synthesized compounds were confirmed from their respective, IR, <sup>1</sup>HNMR, <sup>13</sup>C NMR and Mass studies. From the Antimicrobial screening it was observed that all the compounds exhibited activity against all the organisms employed, the compound shows good antibacterial and antifungal activity.

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