Executive Summary

of

Minor Research Project

entitled

Studies On Some Biologically Active Phenone Derivatives And Their Metal Complexes

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Introduction :

Complex forming (Chelating) agents are becoming of increasing importance in analytical chemistry such as in gravimetric, titrimetric and colorimetric measurements. New types of complexes and complex forming agents are constantly under investigation, for possible analytical and industrial applications. The ever-increasing number of publications on this subject may realize the growing importance of the use of metal chelates in analytical chemistry.

It was G.T. Morgan and Drew57 who first coined the name CHELATE from the Greek word CHELE used for crabs claw to designate to cyclic structures, which arise from the union of metallic ions with organic or inorganic molecules, with two, or more points of attachments to produce a closed ring.

To form metal complexes of an organic compound, an organic compound must have two or more atoms usually oxygen or nitrogen, capable of coordinating with a metal ion, that is, it must be a base having a pair of unshared electrons available for coordination. These coordinating atoms are so arranged that rings of five or six membered including the metal ion will be formed58.

Colored complexes are used for qualitative or quantitative determinations of the elements. For qualitative detection, generally the spot test technique is followed. Whereas in the colorimetric

procedures, based on the formation of colored complexes, utilized for the quantitative determination of elements. Colored complexes like Hemoglobin, Chlorophyll and Vitamin B-12 are also important in biosciences. The development of the spot test techniques for the detection of an organic or inorganic substance is due to the pioneering work of Feigal. Thus it is proper to give outline about formation of complex and complexing agents.

Formation of complexes:

The complex formation and its stability depend on the following three points.

(i) Central metal atom

(ii) Complex forming groups of molecule

(iii) Nature of metal ligand bond

(i) Central metal atom:

The oxidation state of the central metal atom influences to the properties of the metal complex. This can be studied by comparing the complexes formed by the different metal atom in given oxidation state.

(ii) complex forming group of molecules:

The organic molecules can possess a very high ability to form complexes rings. When a molecule functions as a complex forming agent it must fulfill two of the most important conditions given below.

a) The organic molecule must have at least one ore more appropriate functional groups, the donor atoms of which are capable of combining with the metal atom by donating a pair of electrons.
b) The functional group may be an acidic group which may combine with the metal atom by replacement of hydrogen or a coordinating group, permit the ring formation with a metal atom as the closing member.

However, these two conditions are necessary but are not sufficient always for the formation of a complex ring. Steric factors occasionally affect it. The functional group must be appropriately situated in the molecule to influence complexation.

(iii) Nature of the metal ligand bond:

It is necessary to understand the nature of the bond between metal and ligand, for the proper interpretation of the structure of metal complexes. Various theoretical approaches to this problem were developed but it was Jorgensen who proved that the earlier theories proposed by the various authors were fallacious.

Werner proposed his coordination theory for the recognition of the existence of the species such as [PtCl6]2-, [Co (NH3)6]3+ etc. He explained the formation and existence of these species by suggesting that the valency of the atom and the number of bonds it can form may not be identical. He postulated that the combining power of an atom is divided into two spheres of attraction the inner co-ordinate sphere and the outer ionization sphere. Neutral molecules or negative ions are coordinated around the central metal ion in the inner sphere. Number of such groups is the coordination number of the metal ion. Negative ions are loosely attached to outersphere and can be ionizable. So inner-sphere satisfies the secondary valency (non ionizable valency) and outer-sphere satisfies the primary valency (ionizable valency).

Lewis and Langmuir were the first to interpret the nature of the covalent bond as "Sharing of electrons between two bonding atoms in which each atom contributes one electron". Later on Sidgwick64 developed an electronic interpretation to explain the bonding in metal complexes and he had introduced the idea of coordinate bond by accepting the Lewis concept of covalent bond.

Complex forming reagents: (i.e. Ligands)

Prior to the 1980's, research in the field of complex forming reagents (CFRs) was one of the most active research areas in inorganic, analytical chemistry65-66. The development of CFRs was stimulated by research and progress in coordination chemistry and by studies of complex equilibria in solution67. At present the significance of CFR is considered by many as having decreased in favor of instrumental methods, especially for routing, trace and automated analysis, However, CFR remain essential for many current, frequently used methods such as molecular

spectrophotometry in the UV visible region, luminescence analysis and the liquid-liquid extraction of neutral, anionic and cationic species. In addition, CFR are essential in the application of highly efficient separation procedures such as high performance liquid chromatography, pre-concentration of trace elements, a variety of continuous and automated analytical procedures, methods such as AAS, ESR, NMR and some electro analytical methods.

The current research work consists of preparation, recognition and relevance of new α,β – unsaturated propio phenone derivates and its complexies from HCBA, HBBA and HDBA. In the current work, different aromatic were taken for synthesis of from HCBA, HBBA and HDBA. The prepared α,β – unsaturated propio phenone derivates were transformed to its complexies by using Co and Ni metal salts solution with specified PH range. They were given to C, H, N analysis, UV, IR, NMR and MASS spectral analysis for its recognition. The antimicrobial reactivity of synthesized molecules were measured too.

SUMMARY OF RESEARCH WORK CARRIED OUT IN PROJECT :

A. Preparation, Identification and Microbial activity of Phenone derivatives from 1– (4–Benzyloxy-3-Chloro-2-Hydroxy phenyl) ethanone (HCBA) & its Complexes

All chemical reactants used in forming of compounds were Analytical grade reactant quality. In the present chapter, the 1–(4–benzyloxy-3-chloro-2-hydroxy)ethanone (HCBA), α,β – unsaturated propio phenone derivates and they were prepared in good yield and good purity by following the established process. This synthesized compound converted to its Co and Ni metal chelates. The reactions progresses were monitored by TLC.

The prepared compounds were recrystallized and subjected to various analytical techniques for their identifications. The identifications of prepared compounds were done by their C, H, N elemental analysis, UV, IR spectra, NMR spectra and MASS spectra etc. The interpretations of these spectra were given in this chapter.

To find out the biological activities of all prepared compounds, the purified products were screened for their antibacterial and antifungal activity by using cup-plate diffusion method.

Table I:Physical parameters of HCBA and its α,β – unsaturated propio phenone derivates :

Comp. No.	Name of the Compound	Molecular Formula	MW by MASS		Colour	M.P. (^{0}C)	R_f	% yield
			Cal	Fd				
1	1– (4–Benzyloxy-3-Chloro-2-Hydroxy phenyl) ethanone**(HCBA)**	$C_{15}H_{13}ClO_3$	276.71	277	Yellow powder	156	0.88	64.30
1a	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-phenylprop-2-en-1-one	$C_{22}H_{17}ClO_3$	364.22	365	Yellow powder	125	0.90	54.23
1b	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(4-bromophenyl)prop-2-en-1-one	$C_{22}H_{16}BrClO_3$	443.72	444	Yellow powder	195	0.91	56.91
1c	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(4-chlorophenyl)prop-2-en-1-one	$C_{22}H_{16}Cl_2O_3$	399.27	400	Yellow powder	168	0.96	58.32
1d	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one	$C_{23}H_{19}ClO_4$	394.85	395	Yellow powder	135	0.97	61.81
1e	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(3-chlorophenyl)prop-2-en-1-one	$C_{22}H_{16}Cl_2O_3$	399.27	400	Yellow powder	161	0.95	56.50
1f	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(2-chlorophenyl)prop-2-en-1-one	$C_{22}H_{16}Cl_2O_3$	399.27	400	Yellow powder	164	0.92	55.37
1g	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(4-(dimethylamino)phenyl)prop-2-en-1-one	$C_{24}H_{22}ClNO_3$	407.89	408	Yellow powder	143	0.81	57.10
1h	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(3-bromophenyl)prop-2-en-1-one	$C_{22}H_{16}BrClO_3$	443.72	443	Yellow powder	188	0.92	53.25
1i	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(2-hydroxyphenyl)prop-2-en-1-one	$C_{22}H_{17}ClO_4$	380.82	381	dark Yellow powder	181	0.65	53.71
1j	1-(4-(benzyloxy)-3-chloro-2-hydroxyphenyl)-3-(3-hydroxyphenyl)prop-2-en-1-one	$C_{22}H_{17}ClO_4$	380.82	381	Orange Yellow powder	197	0.60	55.33

B. Preparation, Identification and Microbial activity of Phenone derivatives from 1– (4–Benzyloxy-3-Bromo-2-Hydroxy phenyl) ethanone(HBBA) & its Complexes

All chemical reactants used in forming of compounds were Analytical grade reactant quality. In the present chapter, the 1–(4–benzyloxy-3-bromo-2-hydroxy)ethanone (HBBA), α,β – unsaturated propio phenone derivates and they were prepared in good yield and good purity by following the established process. After that synthesized compounds converted to its Co and Ni metal complexies.

The reactions progresses were monitored by TLC. The prepared compounds were recrystallized and subjected to their spectral analysis for their identifications. The identifications of prepared compounds were done by their C, H, N elemental analysis, UV, IR spectra, NMR spectra and MASS spectra etc. The interpretations of these spectra were given in this chapter.

To find out the biological activities of all prepared compounds, the purified products were screened for their antibacterial and antifungal activity by using cup plate diffusion-method.

Table II:Physical parameters of HBBA and its α,β – unsaturated propio phenone derivates :

Comp. No.	Name of the Compound	Molecular Formula	MW by MASS		Colour	M.P. (^{0}C)	R_f	% yield
			Cal	Fd				
2	1– (4–Benzyloxy-3-Bromo-2-Hydroxy phenyl) ethanone**(HBBA)**	$C_{15}H_{13}BrO_3$	321.17	322	Yellow powder	158	0.82	66.23
2a	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-phenylprop-2-en-1-one	$C_{22}H_{17}BrO_3$	409.27	411	Yellow powder	129	0.90	62.33
2b	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(4-bromophenyl)prop-2-en-1-one	$C_{22}H_{16}Br_2O_3$	488.17	488	Yellow powder	198	0.91	61.12
2c	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(4-chlorophenyl)prop-2-en-1-one	$C_{22}H_{16}ClBrO_3$	443.72	444	Yellow powder	170	0.96	64.90
2d	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(4-methoxyphenyl)prop-2-en-1-one	$C_{23}H_{19}BrO_4$	439.30	439	Yellow powder	140	0.97	62.27
2e	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(3-chlorophenyl)prop-2-en-1-one	$C_{22}H_{16}BrClO_3$	443.72	443	Yellow powder	164	0.94	65.10
2f	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(2-chlorophenyl)prop-2-en-1-one	$C_{22}H_{16}BrClO_3$	443.72	443	Yellow powder	166	0.95	63.56
2g	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(4-(dimethylamino)phenyl)prop-2-en-1-one	$C_{24}H_{22}BrNO_3$	452.34	453	Yellow powder	146	0.89	59.94
2h	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(3-bromophenyl)prop-2-en-1-one	$C_{22}H_{16}Br_2O_3$	488.17	489	Yellow powder	190	0.93	63.87
2i	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(2-hydroxyphenyl)prop-2-en-1-one	$C_{22}H_{17}BrO_4$	425.27	425	dark Yellow powder	184	0.86	57.71
2j	1-(4-(benzyloxy)-3-bromo-2-hydroxyphenyl)-3-(3-hydroxyphenyl)prop-2-en-1-one	$C_{22}H_{17}BrO_4$	425.27	425	Orange Yellow powder	198	0.84	53.87