Original Article

Synthesis of Pyrimidine 2-ol/thiol Derivatives of Benzimidazole as a Ligand and Their Bi (III) Metal Complexes by Conventional as Well as Microwave Technique

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ABSTRACT

Objectives: The aim of this investigation is a comparative study between traditional (conventional) and microwave technique of metal complexes of Bi (III) with pyrimidine derivatives as a ligand and also determining their antimicrobial activity against selected organisms.
Method: Pyrimidine-2-ol/thio derivatives of benzimidazole have been synthesized by 2-acetyl benzimidazole with different aromatic aldehydes and finally the product was cyclised with urea and thiourea to form pyrimidine derivatives of benzimidazoles. In this process we have used both the microwave technique as well as conventional technique. It was observed that the reaction which requires 24-30 hours in conventional method is completed in 25-40 minute by MWI technique.
Result: The complexes synthesized through conventional and MWI method were compared in terms of solvent used, time required for synthesis, yield and purity of products. The synthesized ligand and thir metal complexes were characterised through IR, ^{1}HNMR spectroscopy.
Coclusion: On the basis of spectrophotometric determination it may be concluded that the metal complexes of Bi (III) appears to acquire the coordination number five and have distorted octahedral geometry. The antimicrobial activity of free pyrimidine ligands and their metal complexes were determined. These activities were compared with the activity of known antibiotics such as Streptomycin and Fluconazole.

Keywords: Pyrimidine derivatives, Bi (III) chloride, Microwave irradiation, Spectral study.

INTRODUCTION

In pyrimidine ring system two nitrogen atoms are present at position 1 and 3 of the six membered ring. They possess a broad spectrum of biological activities such as antiviral[1,2], anticancer[3,4] antibacterial[5], anti-inflammatory[6], antitubercular[7] and antihypertensive[8]. The pyrimidine nucleus is an important structure in synthetic and medicinal chemistry.

Benzimidazole derivatives are known to posses antifungal, antibacterial, antiviral, anti-inflamattory, antidepressive, and antipyretic activities.[9-12] Thus we become interested in the synthesis of pyrimidines containing benzimidazole moiety by using both the conventional as well as microwave techniques.

Microwave irradiation (MWI) has gained popularity as a powerful nonconventional tool for rapid, efficient and ecofriendly synthesis of a variety of compounds because of selective absorption of microwave energy by polar molecules.[21] The applications of MWI to provide enhanced reaction rate, improved yield and cleaner products is providing quite successful in the formation of a variety of carbon-heteroatom bonds.

The synthesis of heterocyclic organic ligands which preferentially interact with particular metal ions has fundamental importance in many areas of Chemistry. Heterocyclic diazine such as pyrimidine is known to act as bidentate ligand when coordinated with metal ions.[22]

In the present work I have discussed Bi (III) metal complex of pyrimidine derivatives of benzimidazole. The ground state configuration of Bismuth is ns^2p^3 which result in the relative oxidation states of +3(III) and +5(V).

EXPERIMENTAL

All chemicals used were of analytical grade (AR), from Loba, Merk, and SD-Fine, most of them were used as received, without further purification, while some of them were purified and dried through standard procedures, before used throughout the work.

Ethanol (Qualigens, b.p. 78^0C) was refluxed over freshly ignited calcium oxide for 6-8 hours and then distilled, was then fractionated over sodium ethoxide successively. For MORE technique ethanol is more preferred benign organic solvent[13-14] due to its polarity, moderate boiling point, low toxicity, easy recovery, renewability, its biodegradable resource. The domestic microwave oven was used for the synthesis of ligands as well as metal complexes of Bismuth (III).Anhydrous metal chloride was used for complexation under moisture less conditions.

Synthesis of metal complexes

The reaction of benzimidazole substituted pyrimidine derivatives has not been reported so far and pyrimidine derivative may afford good chelating ligand. Thus the present communication comprises the studies onbenzimidazole substituted pyrimidines and their metal complex with Bi (III) chloride.

A new series of novel metal complexes have been synthesized by following techniques.

- **Traditional method** [23]
- **Microwave irradiation method**

Synthesis of Ligand: 4-(benzimidazol-2'-yl)-6-(substituted phenyl) pyrimidine-2-ol/thiol

The following steps are used for synthesis of ligand.

Step (i)

Synthesis of 2-(1-Hydroxyethyl) benzimidazole

Microwave method

The mixture of o-phenylenediamine and lactic acid (1:1) were irradiated at 180 watt for 4-5minute. Reaction mixture was cooled to room temperature and added with 10% NaOH (10 ml) till the solution became slightly basic. The precipitates were separated by filtration and washed with water. **(Compound A)**

Conventional method

The mixture of o-phenylenediamine and lactic acid (1:1) were refluxed on water bath for about 4-6 hour. Reaction mixture was cooled to room temperature and added with 10% NaOH (15 ml) till the solution became slightly basic. The precipitates were separated by filtration and washed with water.[24] **(Compound A)**

Step (ii)

Synthesis of 2-acetyl benzimidazole

For this synthesis only room temperature is required.

To a solution of compound 1 in aqueous acetic acid (5%v/v, 10 ml) was added at room temperature with the solution of potassium dichromate (10 mmol) in aqueous acetic acid (5%v/v, 10 ml) and the mixture was stirred for 15 min. The separated product was filtered, washed with water and dried. The dried product was crystallized from ethanol. This reaction was performed at room temperature.[25] **(Compound B)**

Step (iii)

Synthesis of aromatic aldehyde derivatives of benzimidazole

Microwave method

To a solution of compound 2 (0.01 mol) in aq. NaOH (10%, 20ml) was added with respective aldehyde with proper stirring. The mixture was irradiated for 5-8 minute. Product was cooled and poured into ice cold water then washed with water and recrystallized with ethanol. **(Compound C)**

Conventional method

To a solution of compound B (0.01 mol) in aq. NaOH (10%, 40ml) was added with respective aldehyde at room temperature with proper stirring and refluxed for 6-10 hour.[15-19] The reaction mixture was kept overnight at room temperature. The precipitate was separated, filtered and washed with water and recrystallized with ethanol.[26] **(Compound C)**

Step (iv)

Synthesis of pyrimidine derivative of benzimidazole

Microwave method

A mixture of compound C (0.01 mol) and urea/ thiourea(0.01 mol) in ethanolic sodium acetate (10ml) were irradiated for 8-10 minute. Reaction mixture was cooled and poured into ice cold water and the solid was filtered then washed with water and crystallized from ethanol-benzene (1:1).[27] **(Compound D)**

Conventional method

A mixture of compound C (0.01 mol) and urea/ thiourea (0.01 mol) in ethanolic sodium acetate (30ml) were refluxed on water bath for 5-8 hour. The solvent was evaporated and the residue was

poured into ice cold water. The precipitate was collected by filtration and crystallized from ethanol-benzene (1:1)**(Compound D)**

See Scheme: 1

Synthesis of Metal Complexes

Metal complexes of trichloroBi(III) with selected ligands 4-(benzimidazol-2-yl)-6-(substituted phenyl) pyrimidine-2-ol/ thiol have been synthesized in 1:2 molar ratio of metal and ligand respectively.[28] These metal complexes were synthesized by taking alcoholic solution of dry ligand and dry metal chloride through reported procedures [15-21] using novel MWI as well as traditional method.

Synthesis of Trichloro Bi (III)4-(benzimidazol-2-yl)-6-(substituted phenyl) pyrimidine-2-ol/ thiol in 1:2 ratios

Microwave Method

To a hot methanolic solution (5 ml) of 4-(benzimidazol-2-yl) -6-(3-phenyl) pyrimidine-2-ol/ thiol (0.02 mol) and a hot methanolic solution (5ml) of dry $BiCl_3$(0.01 mol)[28] in 30 ml of methanol were taken in a 100ml borosil conical flask, then reaction mixture was irradiated to microwave oven for 15-20 minutes, till the colour of reaction mixture underwent a change, it was cooled at room temperature and kept overnight. The yellow coloured solid thus obtained was filtered and washed with ethanol. It was dried at room temperature over anhydrous calcium chloride in desiccators, then crystallized from ethanol and dried in vacuum to get purified product. The remaining Bi (III) complexes, with different ligands have also been synthesized from the same route. The physical data of the compounds are summarized in the **table 2.**

Conventional method

To a hot methanolic solution (10 ml) of 4-(benzimidazol-2-yl)-6-(substituted-phenyl)pyrimidine-2-ol/thiol (0.02mol) and a hot methanolic solution (10ml) of dry $BiCl_3$ (0.01mol) in 70 ml of methanol were taken in a 250ml borosil round bottom flask and fitted with water condenser then reaction mixture was refluxed for 4-6 hour till the colour of reaction mixture underwent a change, it was cooled at room temperature and kept overnight. The yellow coloured solid thus obtained was filtered and washed with ethanol. It was dried at room temperature over anhydrous calcium chloride in desiccators, then crystallized from ethanol and dried in vacuum to get purified product. The remaining Bi (III) complexes, with different ligands have also been synthesized from the same route. The physical data of the compounds are given in the **table2.**

$$BiCl_3 + 2(C_{11}H_7N_4XAr) \longrightarrow Bi(C_{11}H_6N_4XAr)_2Cl + 2HCl$$

Here X= O, S

Ar = C_6H_5, 4-$OCH_3C_6H_4$, 3-ClC_6H_4, 4-ClC_6H_4, 4-$NO_2C_6H_4$

RESULT AND DISCUSSION

All the synthesized ligands and their metal complexes were characterized by melting point determination, IR, H^1NMR, Mass spectral study. The molecular weight measurements through LCMS show the monomeric nature of these compounds. All compounds were stable at room temperature. The complexes were crystalline or amorphous, insoluble in water while soluble in ethanol, ether, acetone, chloroform and DMSO.

Present study describes the series of Chlorobismuth (III) pyrimidine-2-ol/ thiolmetal complex. The bidentate behaviour of these ligands has been confirmed by IR, ^{1}HNMR spectral data is given below in **table 3.** The presence of new bands in the comparison of free ligands in the IR region of 425-448 cm^{-1} and 528-548 cm^{-1} shows

stretching vibrations of Bi-N (benzimidazole) and Bi→N (Pyrimidine) respectively. In these complexes the central metal atom bismuth (III) appears to acquire the coordination number five and the most possible geometry around the bismuth atom is distorted octahedral. The structures of metal complexes are given **in Scheme: 1**

The ligands and their Bi (III) metal complex were synthesized in 1:2 molar ratios have been characterized with the help of following spectral data given below in the **table 3.**

All the synthesized ligands and metal complexes were tested their biological activity against selected microorganisms. The results are given in the **table: 4.**[29]

The activity of microbes was measured in terms of zone of inhibition in mm including the well diameter.

- **remarkable activity**= ≥ 18mm,
- **good activity**=14-18mm
- **moderate activity**= 8-14mm,
- **no activity**= 0-8mm

Comparison of the antimicrobial activity of pyrimidine ligands and their metal complexes with standard drug streptomycin and fluconazole exhibited following results: Ligand 1, 2, 3, 5 showed no activity against E. coli but their metal complexes show moderate activity against E. coli. These ligands showed moderate activity against S. pyogens, S. aureus and P. vulgaris but their metal complexes showed good activity against S. pyogens, S. aureus and P. vulgaris. Similarly ligand 4 and 5 showed moderate activity against A. niger and A.clavatatus but their metal complexes showed good activity against A. niger and A.clavatatus.[30]

Similarly Comparison of the antimicrobial activity of ligands and their metal complexes with streptomycin and fluconazole exhibited following results: Ligand 9 and 10 showed good activity against both gram positive and gram negative bacteria but their metal complexes showed excellent activity against both gram positive and gram negative bacteria. On the other hands ligand 8, 9 and 10 and their metal complex showed excellent activity against A. niger and A.clavatatus.

Therefore it can possibly be concluded that the activity of Bi (III) complexes with pyrimidine ligands increased the antimicrobial activity as compared to free ligand. The results are given in above table.

CONCLUSION

The ligands and metal complexes were synthesized through traditional method as well as microwave method. The results were compared in terms of time required for synthesis and solvent used, besides maintaining the conditions of reaction procedure and generation of waste. Most organic reaction requiring heat have been heated using traditional heat transfer equipments such as oil bath, sand bath or heating mantels. These techniques are rather slow and create a temperature gradient within the sample. Microwave assisted reactions[31] have received great interest because of their simplicity in operation, enhanced reaction rates, reduce reaction time, products with high purity and better yields compared to those conducted by conventional heating method.

In this way we found that efficient and convenient procedure for the synthesis of benzimidazole substituted pyrimidine ligands and their Bi (III) metal complexes. Moreover in microwave technique the reactions can be carried out under less solvent conditions which play a strategic role as the solvents are often very toxic, expensive and difficult to use. Hence the use of microwave technique for the synthesis of organic and inorganic compounds will be the part of "**green chemistry**".

Applications of some synthesized metal complexes bismuth with pyrimidine

derivatives have been discussed.All the synthesized compounds are tested by their bactericidal activity against*Escherichia coli,Proteus vulgaris,S. aureus, S.albus, S.pyogens*and also fungicidal activity against*A. niger, C. albicans and A.clavatatus*.The antimicrobial activity of free pyrimidine ligands and their Bi (III) metal complexes in 1:2 molar ratios were examined. These activities were compared with known antibiotics such as Streptomycin and Fluconazole. It is observed that metal complexes showed better activity than the activity of free ligands but lower activity than the activity of standard drugs.

Therefore it may be concluded that the complexation of metal moiety with bioactive pyrimidine ligand increases the biological activity of the complexes.

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Table 1. Synthetic and physical data for 4-(benzimidazol-2-yl)-6-(substituted phenyl) pyrimidine-2-ol/thiol (Ligand)

S. NO.	Ligand	Mol. Weight	Melting Point(^{0}C)	Time Required (in min)		Solvent Used (in ml)	
				Conv.	MWI	Conv.	MWI
1	$C_{17}H_{12}N_4O$	288.30	284	1440	25	100	40
2	$C_{18}H_{14}N_4O_2$	318.32	286	1320	22	85	40
3	$C_{17}H_{11}N_4ClO$	322.76	292	1200	20	90	35
4	$C_{17}H_{11}N_4ClO$	322.72	204	1320	22	95	35
5	$C_{17}H_{11}N_5O_3$	333.43	202	1080	24	100	40
6	$C_{17}H_{12}N_4S$	304.24	273	1080	20	100	40
7	$C_{18}H_{14}N_4OS$	334.44	283	1200	22	90	40
8	$C_{17}H_{11}N_4ClS$	338.68	277	1320	21	85	35
9	$C_{17}H_{11}N_4ClS$	338.80	288	1440	20	95	40
10	$C_{17}H_{11}N_5O_2S$	349.36	296	1320	22	85	35

Table 2. Comparative synthetic and physical data for metal complex of 4-(benzimidazol-2-yl)-6-(substituted phenyl) pyrimidine-2-ol/thiol with Bi (III) in 1:2 molar ratios

S.NO.	Formula	Melting Point(^{0}C)	Time Required (in min)		Solvent Used (in ml)	
			Conv.	MWI	Conv.	MWI
1	$(C_{17}H_{11}N_4O)_2BiCl$	259	300	15-20	85	35
2	$(C_{18}H_{13}N_4O_2)_2BiCl$	285	300	15-20	80	35
3	$(C_{17}H_{10}N_4ClO)_2BiCl$	278	300	15-20	80	30
4	$(C_{17}H_{10}N_4ClO)_2BiCl$	265	300	15-20	80	35
5	$(C_{17}H_{10}N_5O_3)_2BiCl$	252	300	15-20	80	35
6	$(C_{17}H_{11}N_4S)_2BiCl$	255	300	15-20	80	35
7	$(C_{18}H_{13}N_4OS)_2BiCl$	208	300	15-20	80	30
8	$(C_{17}H_{10}N_4ClS)_2BiCl$	298	300	15-20	80	35
9	$(C_{17}H_{10}N_4ClS)_2BiCl$	286	300	15-20	80	35
10	$(C_{17}H_{10}N_5O_2S)_2BiCl$	274	300	15-20	80	35

Table 3. IR,[1]HNMR[32] Spectral data of4-(benzimidazol-2-yl)-6-(substituted phenyl) pyrimidine-2-ol/thiolwith Bi (III) Chloride in 1:2 molar ratio

S. No.	Free Ligand & their Metal Complex	IR Spectra (in cm-1)	[1]HNMR Spectra (ppm)
1	$C_{17}H_{12}N_4O$	3556 v(-OH), 3356 v(-NH),1605 v(C=N), 1066 v(C-N)1500(C=C),906(CH=CH), 3060 v(Ar-nu)	3.44 (s,-NH), 7.28-7.59(m,Ar-CH) 8.02 (C-H) 10.8 (-OH)
	$(C_{17}H_{11}N_4O)_2BiCl$	3550 v(-OH),3320 v(-NH), 1555 v(C=N), 1062 v(C-N), 1502(C=C),902(CH=CH), 445 v(Bi-N), 530 v(Bi→N)	
2	$C_{18}H_{14}N_4O_2$	3566 v(-OH), 3364 v(-NH)1606 v(C=N), 1078 v(C-N), 1232 v(Ar-OCH3)1508(C=C),837(CH=CH),	3.66 (s,NH), 7.25-7.60 (m,Ar-CH) 7.99 (C-H) 10.5 (-OH)
	$(C_{18}H_{13}N_4O_2)_2BiCl$	3555 v(-OH), 3328 v(-NH),1542 v(C=N), 1070 v(C-N), ,1504(C=C),835(CH=CH), 445 v(Bi-N), 530 v(Bi→N)	
3	$C_{17}H_{11}N_4ClO$	3558(-OH),3372 v(-NH), 1608(C=N), 1080 v(C-N), 766 v(Ar-Cl), 1478(C=C),798(CH=CH),	3.59 (s,NH), 7.26-7.68 (m,Ar-CH) 7.86 (C-H) 10.2 (-OH)
	$(C_{17}H_{10}N_4ClO)_2BiCl$	3554(-OH), 3343 v(-NH), 1560(C=N), 1078 v(C-N), 1477(C=C), 815(CH=CH), 442 v(Bi-N),528 v(Bi→N)	
4	$C_{17}H_{11}N_4ClO$	3564(-OH),3375 v(-NH) , 1602(C=N), 1082 v(C-N), 756(Ar-Cl), 1506(C=C), 840(CH=CH),	3.52 (s,NH), 7.21-7.60 (m,Ar-CH) 7.96 (C-H) 10.6 (-OH)
	$(C_{17}H_{10}N_4ClO)_2BiCl$	3562 v(-OH), 3335 v(-NH),1542 v(C=N), 1077 v(C-N), ,1502(C=C),836(CH=CH), 439 v(Bi-N), 529 v(Bi→N)	
5	$C_{17}H_{11}N_5O_3$	3558(-OH), 3378 v(-NH), 1606 v(C=N), 1082 v(C-N), 1521 v(Ar-NO_2), 1420(C=C), 902(CH=CH),	3.58 (s,NH), 7.25-7.66 (m,Ar-CH) 8.00 (C-H) 10.7 (-OH)
	$(C_{17}H_{10}N_5O_3)_2BiCl$	3549 v(-OH),3346 v(-NH), 1546 v(C=N), 1079 v(C-N), 1412(C=C),899(CH=CH), 435 v(Bi-N), 542 v(Bi→N)	
6	$C_{17}H_{12}N_4S$	830 v(-SH), 3350 v(-NH),1600 v(C=N), 1076 v(C-N)1498(C=C),906(CH=CH), 3060 v(Ar-nu)	3.48 (s,-NH), 7.22-7.54(m,Ar-CH) 8.00 (C-H) 3.23 (-SH)
	$(C_{17}H_{11}N_4S)_2BiCl$	822 v(-SH),3321 v(-NH), 1526 v(C=N), 1066 v(C-N), 1500(C=C),902(CH=CH), 435 v(Bi-N), 539 v(Bi→N)	
7	$C_{18}H_{14}N_4OS$	824 v(-SH), 3360 v(-NH)1608 v(C=N), 1072 v(C-N), 1240 v(Ar-OCH3)1500 v(C=C),837(CH=CH),	3.68 (s,NH), 7.22-7.56 (m,Ar-CH) 7.96 (C-H) 3.25 (-SH)
	$(C_{18}H_{13}N_4OS)_2BiCl$	818 v(-SH), 3329 v(-NH),1550 v(C=N), 1060 v(C-N),,1499(C=C),835(CH=CH), 448 v(Bi-N), 538 v(Bi→N)	
8	$C_{17}H_{11}N_4ClS$	827(-SH),3370 v(-NH), 1606 v(C=N), 1082 v(C-N), 766 v(Ar-Cl), 1476 v(C=C),799 v(CH=CH),	3.59 (s,NH), 7.25-7.63 (m,Ar-CH) 7.96 (C-H) 3.22 (-SH)
	$(C_{17}H_{10}N_4ClS)_2BiCl$	822 v(-SH), 3333 v(-NH), 1562 v(C=N), 1076 v(C-N), 1470 v(C=C), 789 v (CH=CH), 436 v(Bi-N), 545 v(Bi→N)	
9	$C_{17}H_{11}N_4ClS$	824 v(-SH),3372 v(-NH) , 1604 v(C=N), 1084 v(C-N),	3.52 (s,NH),

		756(Ar-Cl), 1506(C=C), 848 v(CH=CH),	7.23-7.62 (m,Ar-CH) 8.06 (C-H) 3.26 (-SH)
	$(C_{17}H_{10}N_4ClS)_2BiCl$	814 v(-SH), 3345 v(-NH),1544 v(C=N), 1079 v(C-N), ,1502(C=C),846 v(CH=CH), 429 v(Bi-N), 535 v(Bi→N)	
10	$C_{17}H_{11}N_5O_2S$	821(-SH), 3368 v(-NH), 1602 v(C=N), 1078 v(C-N), 1521 v(Ar-NO2), 1420(C=C), 900(CH=CH),	3.58 (s,NH), 7.26-7.56 (m,Ar-CH) 8.06 (C-H) 3.27 (-SH)
	$(C_{17}H_{10}N_5O_2S)_2BiCl$	810 v(-SH),3336 v(-NH), 1536 v(C=N), 1076 v(C-N), 1412(C=C),895(CH=CH), 425 v(Bi-N),548 v(B→N)	

Table 4. Antimicrobial activity of 4-(benzimidazol-2-yl)-6-(substituted phenyl) pyrimidine-2-ol/thiol with Bi (III) in 1:2 molar ratio

S. No.	Free ligand and their metal complexes	Zone of inhibition (mm)					
		Bactertia				Fungi	
		E.coli	P. vulgaris	S.aureus	S. albus	A.niger	C.albicans
1	Ligand	06	10	12	13	10	08
	Complex	09	15	16	17	12	11
2	Ligand	05	10	13	14	11	09
	Complex	10	15	15	16	14	13
3	Ligand	07	13	14	17	12	11
	Complex	11	15	17	16	15	14
4	Ligand	09	11	12	12	12	12
	Complex	13	14	15	14	15	16
5	Ligand	07	14	14	13	11	10
	Complex	10	18	16	18	17	15
6	Ligand	09	11	10	12	11	13
	Complex	11	13	12	14	13	16
7	Ligand	10	11	10	11	13	14
	Complex	12	14	12	13	15	16
8	Ligand	13	14	15	16	19	20
	Complex	15	16	17	18	23	24
9	Ligand	15	16	17	16	22	24
	Complex	20	19	19	21	25	25
10	Ligand	15	17	16	17	19	20
	Complex	19	20	20	21	21	22
Streptomycin		20	21	23	22	--	--
Fluconazole		--	--	--	--	26	25

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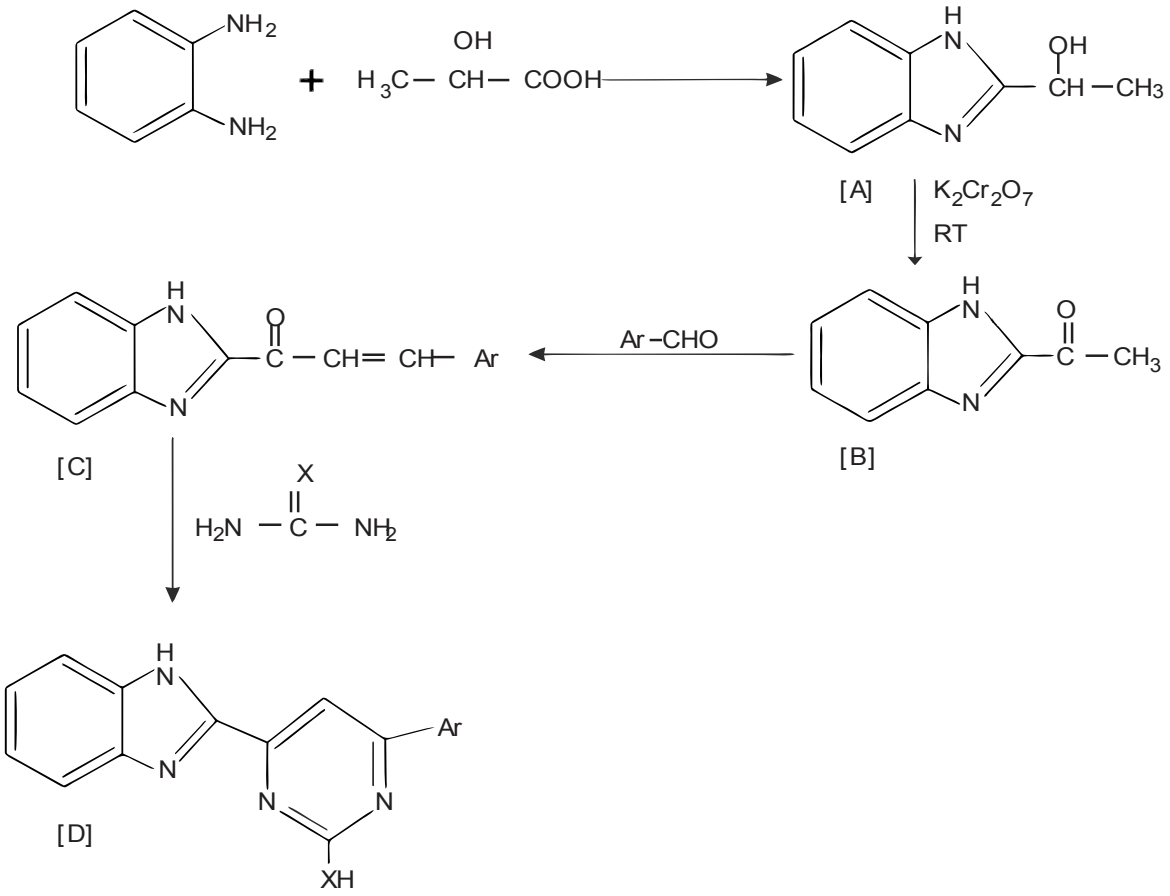

Scheme: 1

Here **X= -O, -S**and **Ar = -C_6H_5, 4-$OCH_3C_6H_4$, 3-ClC_6H_4, 4-ClC_6H_4, 4-$NO_2C_6H_4$**

The physical data of synthesized ligands are given in **table1.**

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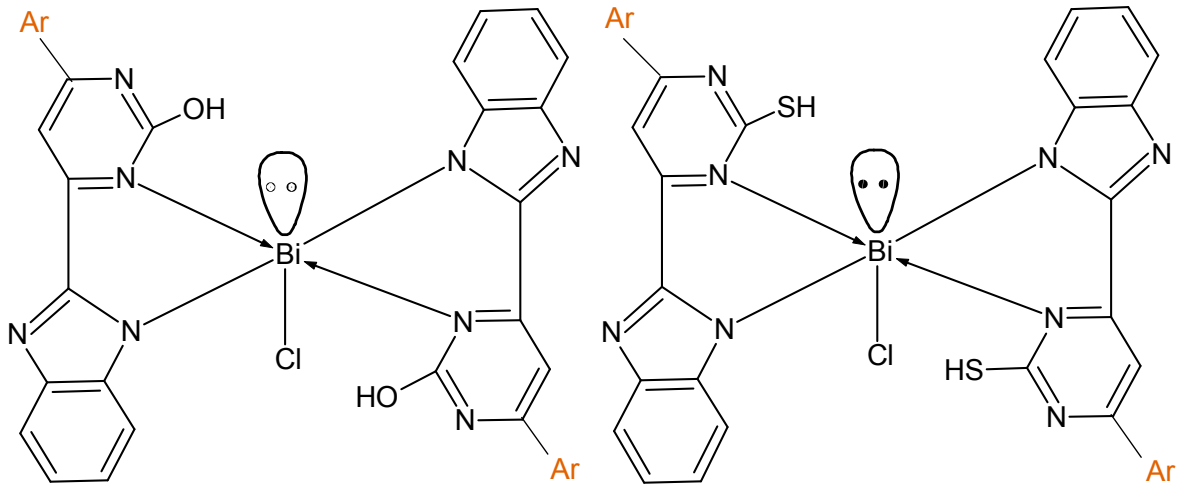

Scheme: 2

Ar = C_6H_5, 4-$OCH_3C_6H_4$, 3-ClC_6H_4, 4-ClC_6H_4, 4-$NO_2C_6H_4$