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Environmentally Benign and Efficient Synthesis of 4-substituted-2-(1-substitutedl-1*H*-Imidazol-5-yl) Naphthalen-1-ol and their antimicrobial Study

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Abstract : New classes of substituted imidazole were efficiently synthesized by using chief, easily available and cost effectiveiodine as a catalyst. 4-Substituted-2-(1-Substitutedl-1*H*-Imidazol-5 yl)naphthalen-1-ol (2)and synthesis of other different substituted imidazole were carried out by cyclising1-[2-(1-hydroxy-4-Substitutednaphthalen-2-yl)-2-oxoethyl]-3-substitutedthiocarbamide (1) in an environmentally benign PEG 400 solvent medium using molecular iodine as an efficient cyclising agent. The reaction completed in short period of time and isolation of product becomes very easy in PEG 400 medium. The scheme outlined above comes under green chemistry parameter.Structure of all the synthesized compounds have been characterized by C, H, N elemental analysis, IR ,^{1}H NMR and Mass spectral analysis.

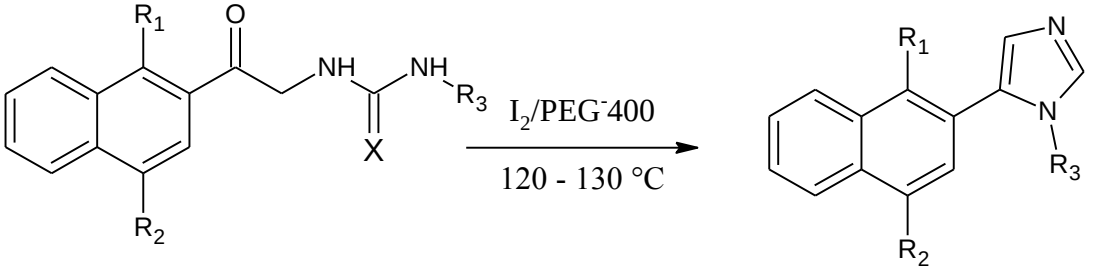
Keywords : Iodine, PEG 400, environmentally benign, efficient, cost effective, cyclising agent..

Introduction

Substituted Imidazolesare the most important pharmacologically and biologically activeheterocyclic compounds. This aromatic heterocyclic is a"1, 3-diazole" and is classified as an alkaloid. Imidazole is nothing but a parent compound, whereas imidazoles are a class of heterocycles containing nitrogen as a heteroatom with similar ring structure, but varying substituents. Imidazole compounds show versatile biological properties such as angiotensin inhibitors[1],antiviral[2], anti-inflammatory [3], inhibitors of p38 MAP Kinase[4], B-Raf kinase[5], HIV-1 protease[6], calcium antagonist and inhibitors of thromboxane A2 synthesase[7], antihistaminic[8], antimuscarinic[9], antiarthritic[10],cardiotonic[11], and antitumor agents[12].Due to such versatile application of imidazole in Pharmaceuticals, Agriculture and Textile industry, the synthesis of these imidazole derivatives has attracted much attention in organicsynthesis. Imidazole was first synthesized by Heinrich Debus in 1858this synthesis, while producing relatively low yields, is still used for creating C-substituted imidazoles. Recently some methods for synthesis of substituted imidazoles are reported silica-bonded propylpiperazine N-sulphamic acid (SBPPSA),[13]$BF_3.SiO_2$,[14] and sulphonated carbon/silica functionalized Lewis acids.[15]silica-supported Wells–Dawson acid,[16]mercaptopropylsilica (MPS),[17]heteropolyacids,[18] by hetero-coperearrangement,[19] condensation of imines and acid chlorides,[20] and from diazocarbonyls by N–H insertion reactions of primary ureas.[21] solid acid nano-catalyst,[22] HClO4-SiO2,[23] MCM-41 or p-TsOH,[24]ZrCl4,[25] DABCO,[26] microwave irradiation,[27]from above literature survey it reveals that the synthesis of imidazole were not carried out in PEG 400 medium using iodine as a catalyst.Also, some of these previous methods have suffered from one or more drawbacks like high temperature requirement, use of hazardous catalyst, toxic and expensive solvent medium that are hazardous to human health and to our delicate environment. Therefore it is necessary to develop new alternative path

avoiding toxic and hazardous catalyst to synthesize the valuable heterocyclic compounds like imidazole. In our present research work we were synthesizes the new substituted derivative of imidazole using chief and easily available and non toxic catalyst such as Iodone and non toxic solvent medium such as Polyethylene glycol 400 when we use such a non toxic chief easily available solvent like PEG 400 and efficient cyclysing catalyst iodine then green chemistry parameter will be maintained.

The synthesis of 4-substituted-2-(1*H*-imidazol-5-yl) naphthalen-1-ol **(**(2) were carried out by cyclization of 1-[2-(4-Substituted-1-hydroxynaphthalen-2-yl)-2-oxoethyl] thiocarbamide (1) in PEG 400 Medium using iodine as a cyclising catalyst **(Scheme -1)**



(1) **Where,** (2)

X= S, O, R_1= H, OH, R_2= H, Br, NO_2, R_3= H, Ph

Scheme-1

Experimental

Materials and Methods

All chemicals used were of AR grades. The melting points of all the synthesized compounds were recorded using hot paraffin bath. The Carbon and Hydrogen analysis was carried out on Carlo-Ebra 1106 analyser. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on Lambda Scientific Pvt Ltd spectrometer in the range 4000-400 cm^{-1} in KBr pellets. PMR spectra were recorded on Brucker AC-500F spectrometer with TMS as internal standard using $CDCl_3$ and DMSO-d_6 as solvent. The purity of compound was checked on silica Gel-G plates by TLC with layer thickness of 0.3 mm.

Procedure for synthesis of 4-bromo-2-(1-phenyl-1H-Imidazol-5-yl) naphthalen-1-ol (2a)

The compound 1-[2-(4-bromo-1-hydroxynaphthalen-2-yl)-2-oxoethyl]-3-phenylthiocarbamide (1a) and iodine (1:25% mol) were taken in 20ml PEG-400 in a 100 ml round bottom flask then refluxed the reaction mixture in an oil bath at 120-130^{0}C for 7 hour the completion for reaction was monitored by TLC (ethylaceate : N-hexane = 50:50) the brown red coloured reaction mixture was poured into saturated solution of sodium thiosulphate, the separated solid product was filtered and washed with distilled water dried and recrastalised from ethyl alcohol.

The yield of the dried crude product was found to be 0.90 g (90%).
Melting Point: - 92-93^{o}C
Colour of compound (2a) - Yellow Crystalline solid
Analysis: Calculated for$C_{19}H_{13}BrN_2O$: C,62.48%; H,3.69%; Br,21.88%; N,7.67%. Found: C,62.50%; H,4.67%; Br,21.90%; N,7.85%.IR (KBr, cm^{-1}):3583.09 cm^{-1},3151.12 cm^{-1},1072.23 cm^{-1},1681.62 cm^{-1},1245.79 cm^{-1},3043.12 cm^{-1},1446.35 cm^{-1}.^{1}H NMR (500 MHz, CdCl3): δ 4.85 (s, 1H, OH), δ 4.466 (s, 1H, C=C-H vinyl),δ 2.88 (s, 1H, -N-CH),δ 7.26 -8.49 (m, 5H,-C10H5)

Procedure for Synthesis of 2-(1H-imidazol-5-yl)-4-nitronaphthalen-1-ol(2b)

The compound1-[2-(1-hydroxy-4-nitronaphthalen-2-yl)-2-oxoethyl]thiocarbamide(1b) and iodine (1:25% mol) were taken in 20ml PEG-400 in a 100 ml round bottom flask then refluxed the reaction mixture in

an oil bath at 120-130^{0}C for 7 hour the completion for reaction was monitored by TLC (ethylaceate : N-hexane = 50:50) the brown red coloured reaction mixture was poured into saturated solution of sodium thiosulphate, the separated solid product was filtered and washed with distilled water dried and recrastalised from ethyl alcohol.

The yield of the dried crude product was found to be 0.85 g (85%).
Melting Point: - 293^{o}C
Colour of compound (2b) - Yellow solid
Analysis: Calculated for$C_{13}H_9N_3O_3$: C,61.18%; H,3.55%; N,16.46%.Found: C,62.23%; H,3.90%; N,16.98%.IR (KBr, cm^{-1}):3548.38cm^{-1},3324.68 cm^{-1},1662.34 cm^{-1},1581.34 cm^{-1},1400.07 cm^{-1},1157.08 cm^{-1},3012.27 cm^{-1},1400.07 cm^{-1}.^{1}H NMR (500 MHz, CdCl3): δ 2.79 (s, 1H, OH), δ 2.67(s, 1H, NH), δ 2.36 (s, 1H, -N-CH), δ 7.51-8.75(m, 5H,-C10H5)

Procedure for synthesis of 4-amino-2-(1H-Imidazol-5-yl) naphthalen-1-ol (2c)

The compound 1-[2-(4-amino-1-hydroxynaphthalen-2-yl)-2-oxoethyl]thiocarbamide (1c) and iodine (1:25% mol) were taken in 20 ml PEG-400 in a 100 ml round bottom flask then refluxed the reaction mixture in an oil bath at 120-130^{0}C for 8 hour the completion for reaction was monitored by TLC (ethylaceate : N-hexane = 50:50) the brown red coloured reaction mixture was poured into saturated solution of sodium thiosulphate, the separated solid product was filtered and washed with distilled water dried and recrastalised from ethyl alcohol.

The yield of the dried crude product was found to be 0.90 g (90%).
Melting Point: - 209^{o}C
Colour of compound (2c) - Yellow Crystalline solid
Analysis: Calculated for$C_{13}H_{11}N_3O$: C,69.32%; H,4.92%; N,18.66%.Found: C,70.32%; H,5.18%; N, 18.50%.IR (KBr, cm^{-1}):3529.09 cm^{-1},3185.83 cm^{-1},3301.54 cm^{-1},1616.06 cm^{-1},1130.08 cm^{-1},782.96 cm^{-1},3004.55 cm^{-1},1430.92 cm^{-1}.^{1}H NMR (500 MHz, CdCl3): δ 5.58 (s, 1H, OH), δ 3.25(s, 1H, NH), δ 2.99(s, 1H, -N-CH), δ 3.22 (s, 2H, Ar-NH_2),δ7.17 -8.91(m, 5H,-C10H5)

Table 1: Synthesis of different substituted imidazole derivative.

Sr No.	Expt. No.	Compound	Yield %	M.P ^{0}C	Colour
1	4	2-(1*H*-imidazol-5-yl)naphthalen-1-ol (2d)	80%	122	Yellow solid
2	5	1-(1*H*-imidazol-5-yl)naphthalen-2-ol (2e)	82%	78	light Yellow solid
3	6	1-(1*H*-imidazol-5-yl)-6-nitronaphthalen-2-ol (2f)	75%	280	Yellow
4	7	6-amino-1-(1*H*-imidazol-5-yl)naphthalen-2-ol (2g)	85%	145	Brown
5	8	6-bromo-1-(1*H*-imidazol-5-yl)naphthalen-2-ol (2h)	90%	187	Light brown
6	9	5-(naphthalen-1-yl)-1*H*-imidazole (2i)	92%	-	Dark brown sticky semi solid
7	10	5-phenyl-1*H*-imidazole(82)	95%	98	Light yellow solid

Antimicrobial Screening

From the above synthesized Imidazole compounds some of them were screened in vitro for their bactericidal activity against Gram positive bacteria (*Staphylococcus aureus*) and Gram negative bacteria (*Escherichia Coli, Salmonella Typhi, Klebsiella Pneumoniae and Shigella dysentariae*), and for their fungicidal activity against *Aspergillus niger and Candida albicans*.

The antimicrobial screening of the above compounds were carried out by agar-well diffusion method. In this method the antimicrobials are allowed to diffuse out into the medium and interact in a plate freshly seeded with the test organisms. The resulting zones of inhibition will be uniformly circular as there will be a confluent lawn of growth. The diameters of zone of inhibition can be measured in millimeters.

Table 2:Showing the activity of synthesized compounds against different Fungal and Bacterial species.

Compound	Activity																				
	Candida			**Aspergillus**			**Salmonella**			**Shigella**			**E.coli**			**S.Aureus**			**Klebseilla**		
	10 µl	20 µl	30 µl	10 µl	20 µl	30 µl	10 µl	20 µl	30 µl	10 µl	20 µl	30 µl	10 µl	20 µl	30 µl	10 µl	20 µl	30 µl	10 µl	20 µl	30 µl
2a	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2b	-	Y	Y	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
2c	-	-	-	-	-	-	Y	Y	Y	Y	Y	Y	Y	Y	Y		-	-	Y	Y	**Y**
2d	-	-	-	Y	Y	Y	Y	Y	Y	-	-	-	-	-	-	-	-	-	Y	Y	**Y**

3. Results and Discussion

Recently, Due to low toxicity, ready availability and easy handling the molecular iodine have been used extensively inorganic synthesis as an efficient cyclising agent. To maintain green chemical approach theSynthesis of 4-bromo-2-(1-phenyl-1*H*-imidazol-5-yl) naphthalen-1-ol (2a)from1-[2-(4-bromo-1-hydroxynaphthalen-2-yl)-2-oxoethyl]-3-phenylthiourea (1a) and iodine (1:25% mol) were carried out inPEG 400 and other various available solvent medium. When we use the medium other than PEG 400 the time required for completion of reactions is in between 8 to 18hours. The solvent medium other than PEG emits pollutant; also these are costly and not easily available, and as well as these solvents are very hazardous to our delicate environment and to the human health. To reduce long time duration of reaction and also for maintaining the green chemistry parameters and to develop new reaction conditions, The reactions were carried out in various solvent mediums and it was observed that the time required to complete the reaction in Polyethylene glycol 400 medium is reduced as compared to the other medium as well as yield also increased as shown in Table 3

Table 3:Reaction of 1-[2-(4-bromo-1-hydroxynaphthalen-2-yl)-2-oxoethyl]-3 phenylthiocarbamide(1a) and iodine

Sr. No.	Medium	Quantity of medium (ml)	Time Duration in hours.	Yield (%)	M.P (^{0}C)
1.	Acetone	30	15 hrs.	54%	92-93^{0}C
2.	Ethanol	25	18 hrs.	56%	94^{0}C
3.	DMF	30	10 hrs	65%	91^{0}C
4.	PEG-400	25	7 hrs.	90%	92-93^{0}C
5.	Acetonitrile	40	24 hrs.	45%	93^{0}C
6.	Iso propyl alcohol	35	20 hrs.	50%	92^{0}C

From above results it was clear that the PEG 400 medium produce the product in good yield in a short period of time, the quantity required is also less among all the reaction conditions. If the reaction performed in this medium then green chemistry will be maintained. Hence author is interested to reduce time span and quantity of solvent used as medium and also to increase yield and to maintain eco-friendly reaction conditions. Physical study and mechanisms were not studied during reactions.

Interactions of different N-substituted and1-[2-(4-substituted-1-hydroxynaphthalen-2-yl)-2-oxoethyl]thiocarbamide(1) and Iodine as catalyst in Polyethylene glycol 400 medium were carried out and the time required for completion of reactions is in between 8 to 12hours. To reduce time duration of reaction and

for maintaining green chemistry parameters and to develop new reaction conditions. The reactions were carried in Polyethylene glycol 400 and in this medium it was observed that time duration of this reaction is reduced, the quantity of medium required is also less and yield is also good.

Table 4: Effect of catalyst concentration on synthesis of 4-bromo-2-(1-phenyl-1*H*-imidazol-5-yl) naphthalen-1-ol (2a)

Sr. No.	Catalyst mol %	Time Duration in hours.	Yield (%)
1.	10	11	15
2.	15	9	40
3.	20	15	65
4.	25	7	85
5.	30	9	75
6.	50	18	72

To study the exact concentration of catalyst required to complete the reaction in good yield the interaction of 1-[2-(4-bromo-1-hydroxynaphthalen-2-yl)-2-oxoethyl] thiocarbamide (1a) with different quantity of Iodine in PEG400 medium were carried out. It is observed that as the quantity of Iodine increases the yield also increased at sufficient level. With further increasing the quantity of catalyst the yield of the product does not increases satisfactorily also the time required is long. So 25% mol of iodine catalyst is the appreciable quantity for this reaction as shown in the above table.

Conclusions

We have developed an efficient PEG promoted solvent and Iodine catalysed method for the synthesis of mono and di-substituted Imidzoles with good yield. These results further demonstrate the importance of PEGpromoted synthesis in avoiding hazardous organic solvents and toxic catalysts with comparatively less reaction time which is in the context green chemistry.

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