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Synthesis Of 1-(4-Chlorophenyl)-2-(3-Sulphoxyphenyl-4-(4-Substituted Benzylidene)-5-Imidazolones

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ABSTRACT
Now a day imidazolones have attracted great attention of chemists due to interesting properties shown by them. They are believed to be associated with several pharmacological activities. Their significance lies in the fact that they show diverse biological activities. Which include anticancer, anticonvulsant, antiparkinsonian, CNS depressant, antimicrobial, antihelmintic, anti HIV, anti-inflammatory etc.However no work is reported on imidazolone from substituted benzoyl glycine.Therefore it was thought interesting to attempt synthesis of imidazolones containing new substituent. In the proposed work, we have reported eight newly synthesized sulphoxy substituted imidazolones from oxazolones and 4-chloroaniline in presence of zeolite as a catalyst. The oxazolones were obtained from 3-sulphoxy benzoyl glycine and variedly substituted aromatic aldehydes in presence of anhydrous sodium acetate and acetic anhydride. The characterisation of these compounds was made by chemical properties, elemental analysis and spectral data like IR, ^{1}H-NMR.The use of zeolite as a catalyst enabled us to reduce reflux time and increase percentage yield of the products.

Keywords: *Sulphoxy benzoyl glycine,oxazolones,Zeolite catalyst ,5-imidazolones.*

INTRODUCTION
Ruhemann and Cunnington [1]reported the first synthesis of 5-imidazolone in 1889 by the condensation of ethyl phenyl propiolate with benzanidine hydrochloride in the presence of sodium ethoxide and obtained 2-pheyl-4-benzylidene-5(4H)- Imidazolone. Imidazolones form an important class of heterocyclic compounds since they can be converted into amino acid [2-3],used in drugs[4],pigments and electrodes[5].They have also shown diverse bioactivities including anticancer[6], anti HIV[7], antiparkinsonian[8-9], CNS depressant[10], antihelmintics[11]. Gabillet S, et al reported A Phosphine-Catalyzed Preparation of 4-Arylidene-5-imidazolones [12]. Snehalatha P and Subhashini N. J. P carried out, "Synthesis, characterization and biological evaluation of novel imidazolones derived from azlactones[13].Ming and coworkers[14] synthesized 2-alkoxy 4H-imidazole-4-ones from aza-witting reaction of iminophosphorane with phenyl isocyanate to form carbodiimide which on subsequent reaction with ROH in presence of RONA gave the target compounds. Kedar and Dehmukh[15]reported "synthesis of 1-(-4-methyl phenyl)-2-(3-bromo phenyl) -4-(4-substituted benzylidene) -5-imidazolones. Chopra et al [16] carried out Microwave assisted synthesis of some 5-substituted imidazolone analogues as a new class of nonpurine xanthine oxidase inhibitors. However no work is reported on imidazolones synthesized from substituted benzoyl glycine. Thus, due to their diverse applications and also part of our study, it was thought interesting to synthesize some new imidazolones containing different substituents.

MATERIALS AND METHOD
Aromatic aldehydes, benzoylglycine, 4-chloroaniline,sodium acetate, acetic anhydride and zeolite are required chemicals purchased from sd fine chemicals. All the chemicals used were of AR grade. Melting points were measured in open capillary tube. The purity of the compounds were checked by TLC on silica gel in petroleum ether and ethyl acetate (80:20).The IR spectra were recorded on Agilent Technologies cary 630 FTIR.^{1}H-NMR spectra were recorded on Brucker AVANCE 400MHz spectrometer using TMS as internal standard. This work involved condensation of 3-sulphoxy benzoyl glycine with substituted aldehyde in acetic anhydride in presence of anhydrous sodium acetate to obtain variedly substituted oxazolones.Oxazolones were further reacted with 4-chloro aniline in presence of zeolite as a catalyst to obtain the target compounds 1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-substituted benzylidene)-5-imidazolones.

Step-I: Synthesis of 2-(3-sulphoxyphenyl)-4-(4-methoxybenzylidene)-5-oxazolone.

To the solution of 3-sulphoxy benzoyl glycine 2.14gm(0.01mol)in acetic anhydride ,added 1.36gms of anisaldehyde(0.01mol)and then .0.7gms anhydrous sodium acetate (0.01mol).The contents were dissolved in ethyl alcohol .The solution was refluxed under water condenser for 3-hours It was poured to ice cold water when an yellowish solid was obtained. Washed it 2-3times with ice cold water and recrystallised from ethanol.

Yield: 65% melting point: 145^{0}C

Molecular formula $C_{17}H_{12}NO_5$ Molecular weight: 342

IR (KBr) in cm^{-1}:3045 (Ar,C-Hstr);2937(Aliph,C-Hstr);1787(C=Ostr);1651(C=Nstr);1596(C=Cstr);1311(C-O bend);1263(C-N bend);1028(S-O Asymmetric str);981(S-O symmetric str).

^{1}H-NMR(∂) : 8.27(d,2H,Ar-H);8.11(d,2H,Ar-H);7.66-7.68(t,1H,Ph-CH);7.58-7.61(t,2H,Ar-H);7.60(d,2H,Ar-H);3.87(s,3H,-OCH_3). Elemental analysis for $C_{17}H_{12}NO_5$ (342)

Calculated	%C=59.64	%H=3.50	%N=4.09	%S=9.35
Found	%C=59.60	%H=3.48	%N=4.02	%S=9.30

Step-II: - Synthesis of 1-(4-clorophenyl)-2-(3-sulphoxyphenyl)-4-(4-methoxybenzylidene)-5-imidazolone

To the ethanolic solution of oxazolone(3.42gm,0.01M) obtained in step-I, added 4chloro aniline (1.27gm,0.01M)and zeolite (1gm) as a catalyst followed by 1ml of 2%aq NaOH solution. The reaction mixture was reflux under water condenser for two and half hours it was allowed to cool and poured to ice cold water obtained colourless solid on acidification with dil HCl Washed it 2-3times with cold water and recrytalised from ethanol

Yield: 60% Melting point: 235^{0}C

Molecular formula $C_{23}H_{16}N_2SClO_4$ Molecular weight: 451

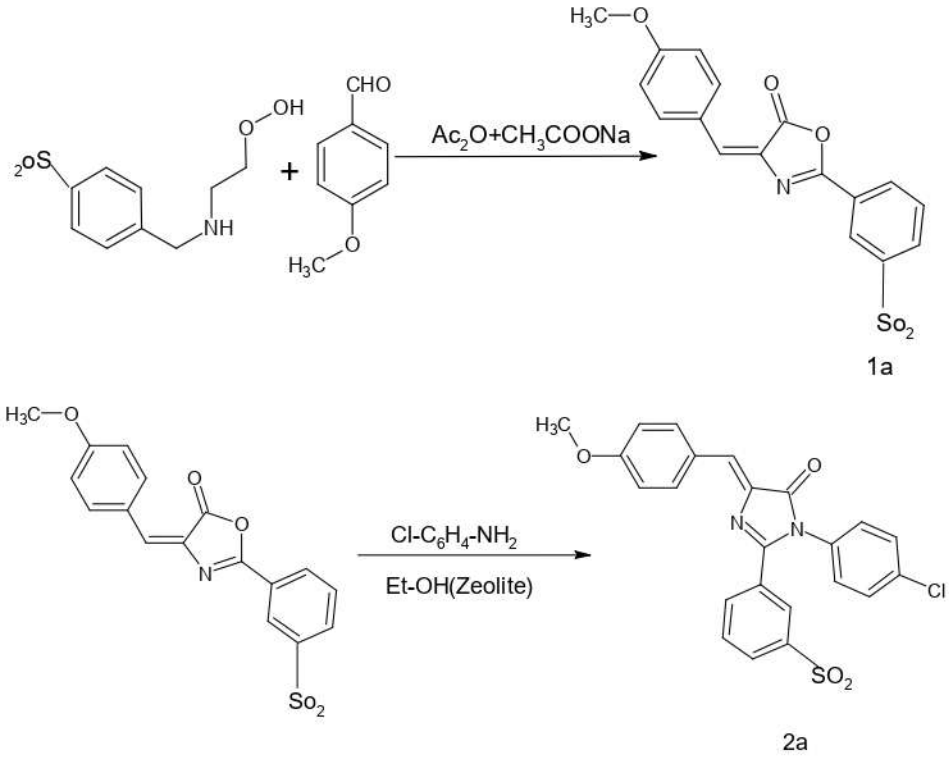
IR (KBr) :3030cm^{-1}(Ar,C-Hstr);2925cm^{-1}(Aliph,C-Hstr)1710cm^{-}

^{1}H-NMR(∂) : 8.04(d,2H,Ar-H);7.74(d,2H,Ar-H);7.56-7.61(m,3H,Ar-H);7.48.-7.52(t,1H,Ph-CH);7.29(d,2H,Ar-H);7.14(S,1H,Ar-H);6.92(d,2H,Ar-H);3.78(S,3H,OCH3)

Elemental analysis for $C_{23}H_{16}N_2SClO_4$ (451.5)

Calculated	%C=61.12	%H=3.54	%N=6.20	%S=7.08	% Cl =7.86
Found	% C=61.05	%H=3.50	% N=6.18	%S=7.02	% Cl =7 .78

REACTION



RESULTS AND DISCUSSION

The oxazolones required for the synthesis of imidazolones were prepared by condensation of newly synthesied 3-sulphoxy benzoyl glycine with variedly substituted aldehydes in presence of anhydrous sodium acetate in acetic anhydride Their formation was confirmed by physical and chemical tests. Thus eight variedly substituted oxazolones were obtained from different aldehyde and 3-sulphoxy benzoyl glycine. The target compounds 1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-substituted arylidene)-5-Imidazolones were synthesized by reacting each of these oxazolones with p-chloroaniline in ethanolic medium in presence of zeolite as a catalyst. It offered easy to workout methodology and also reduced reflux time to two and half hours. The compound (2a) gave positive test (orange colour)with ethanolic solution of 2,4dinitrophenyl hydrazine in presence of cone H_2SO_4 confirming the presence of (2a). It showed absorption bands at 2925cm-1due to Aliphatic ,C-H str;1710cm^{-1}due to C=Ostr;1639cm^{-1} C=N str;1035-1025cm^{-1}S-O Asymmetric str and symmetric str 690 C-Cl str similarly ^{1}H-NMR spectrum of 2 (a) showed the following chemical shift(∂)8.04(d,2H,Ar-H);7.74(S,1H,Ar-H);7.56-7.61(m,3H,Ar-H);7.48-7.52(t,1H,Ph-CH);7.29(d,2H,Ar-H);7.14(S,1H,Ar-H);6.92(d,2H,Ar-H);3.78(s,3H,O-CH_3) Which tallies with Molecular formula of target compound 2a $C_{23}H_{16}N_2SClO_4$ Elemental analysis also supported this formula experimental value of which are comparable with calculated ones all these evidences support the formation of 2(a) similarly other target compounds were synthesized by employing above mentioned procedure.

Table-1: List of synthesized compounds, their % yield and melting points.

Sr. No.	Compounds	%Yield	Melting Point (in ^{0}C)
1	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-methoxy benzylidene) - 5-Imidazolone.	60	235
2	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-	65	290

	nitrobenzylidene) - 5-Imidazolone.		
3	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-hydroxybenzylidene) -5-Imidazolone.	68	220
4	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-dimethylaminobenzylidene)-5-Imidazolone.	62	310
5	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(3, 4,5tri methoxybenzylidene)-5-Imidazolone.	64	170
6	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-chlrobenzylidene) - 5-Imidazolone.	69	230
7	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(2-nitro benzylidene) - 5-Imidazolone.	69	210
8	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-hydroxy3-methoxybenzylidene)-5-Imidazolone.	70	160
9	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-benzylidene) - 5-Imidazolone.	63	195
10	1-(4-chlorophenyl)-2-(3-sulphoxyphenyl)-4-(4-furanylidene) - 5-Imidazolone.	65	200

CONCLUSION

Hence we could synthesize a new series of imidazolones by introducing sulphoxy group as one of the new substituents. Use of zeolite as a catalyst afforded us increase in percent yield and reduction in reflux time. Most of these compounds are expected to show antimicrobial activity. Therefore, it may be suggested that more series of compounds are needed to be synthesized by introducing new substituents on benzylidene moiety in order to enhance its value as a drug.

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