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Studies on Novel Bisaryl Hydrazino-*s*-triazine Derivatives - Part 2

J. A. CHAUDHARI*, R. P. PATEL[#] and M. V. HATHI[†]

*Shri R. K. Parikh Arts & Science College, Petlad-388450. Gujarat, India.

[#]C. U. Shah Science College, Ahmedabad-380014. Gujarat, India.

[†]R. R. Mehta College of Sci. & C. L. Parikh College of Commerce, Palanpur-385001. Gujarat, India.

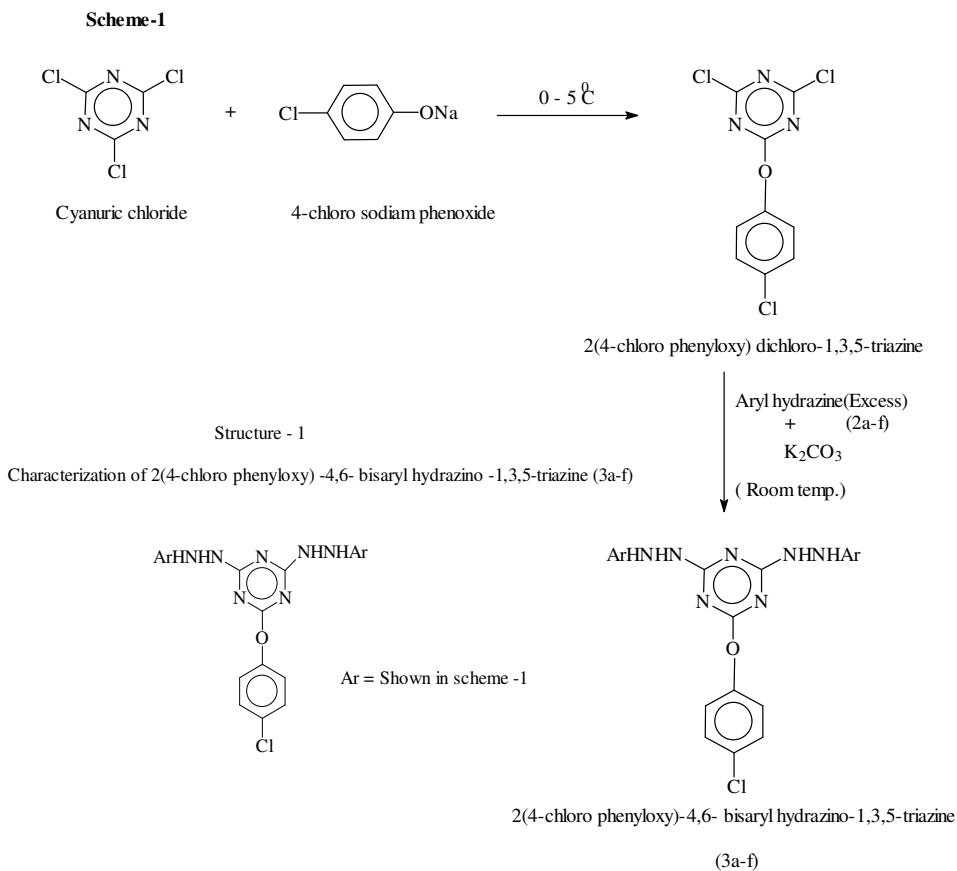
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Abstract: Various 2(4-chloro phenyloxy)-4,6-bisarylhydrazino-1,3,5-triazines(3a-f) were prepared by reaction of 2(4-chloro phenyloxy)-4,6-dichloro -1,3,5-triazine and various aryl hydrazine derivatives. All the 3a-f derivatives were characterized by elemental analysis and IR spectral studies. All the compounds were screened for microbial activity against gram-positive and gram-negative bacteria.

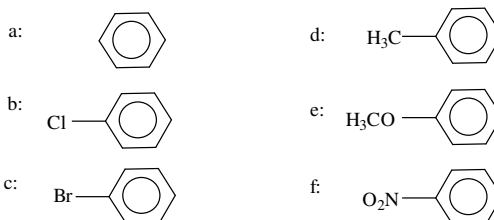
Keywords : 2(4-Chloro phenyloxy) bisarylhydrazino *s*-triazine derivatives, Hydrazines, IR and Spectral study, Antimicrobial activity.

Introduction

Number of derivatives containing *s*-triazine ring have been reported as hetrocyclic compounds¹. They are applicable mostly as reactive dyes and some are used as polymers and drugs^{2,3}. The aryl hydrazine derivatives containing *s*-triazine ring are not reported so far except one instance⁴. Recently our university scientists have studied the hydrazine triazine clubbed molecules having alkoxy group⁴. In extension of this work⁴, the present authors reported the novel hydrazine-*s*-triazine compounds⁵. In continues of this work the present communications deals with the studies on novel *s*-triazines derivatives having the route shown in scheme-1.



Where, Ar :



Experimental

Cyanuric chloride and all the aryl hydrazine derivatives (substitution shown in scheme-1) were obtained as Analar grade from local dealer. 2(4-Chloro phenyloxy)-4,6-dichloro-1,3,5-triazine was prepared by reported method⁴. All other chemicals used were of laboratory grade.

Synthesis of 2(4-chloro phenyloxy) bisaryl hydrazino-s-triazines

2(4-Chloro phenyloxy)-4,6-bis(aryl hydrazino)-1,3,5-triazines (3a-f). The general procedure for these compounds is as follows.

To a well stirred solution of 2(4-chloro phenyloxy)-4,6-dichloro-1,3,5-triazine (0.01 mole) in tetrahydrofuran (THF) solvent (50mL) a solution of aryl hydrazine (or substituted

aryl hydrazine) (scheme-1) (0.02mole) in THF (50mL) was added gradually at room temperature. The mixture was stirred for two hours. Then the mixture was refluxed for further two hours. The resultant solid product was filtered, washed with THF and air-dried. All the (3a-f) compounds listed in Table-1 are dark yellow amorphous powders. Their molecular weights are shown in Table-1.

Table 1. Analysis of synthesized compounds

Compound	Molecular Formula	Mol. Weight	% C		% H		% N	
			Cald.	Found	Cald.	Found	Cald.	Found
3-a	C ₂₁ H ₁₈ N ₇ ClO	419.5	60.07	60.0	4.29	4.2	23.36	23.3
3-b	C ₂₁ H ₁₆ N ₇ Cl ₃ O	488.5	51.59	51.5	3.28	3.2	20.06	19.9
3-c	C ₂₁ H ₁₆ N ₇ ClBr ₂ O	577.5	43.64	43.6	2.77	2.7	16.97	16.9
3-d	C ₂₃ H ₂₂ N ₇ ClO	447.5	61.68	61.6	4.92	4.8	21.90	21.8
3-e	C ₂₃ H ₂₂ N ₇ ClO ₃	479.5	57.56	57.5	4.59	4.5	20.44	20.3
3-f	C ₂₁ H ₁₆ N ₉ ClO ₅	509.5	49.46	49.0	3.14	3.0	24.73	24.6

Mesurements

The elemental analyses of all (3a-f) compounds were determined by TF flash EA. The FT-IR spectra of (3a-f) compounds were scanned in KBr pallets on Perkin Elmer FT-IR spectrophotometer. The NMR spectra of soluble sample No.3e was scanned on Perkin Elmer FT-NMR spectrophotometer.

Antimicrobial Activity

For the testing antimicrobial activity various microorganism were used for the study. The pour plate agar method was used for this study. Following general procedure is adopted⁷.

The antimicrobial activities of all the compounds were studied at 1000 ppm concentration *in vitro*. The different types of microorganism used were some gram negative bacteria (*Escherichia coli*, *Proteus vulgaris*), gram positive bacteria (*Bacillus cereus*, *Streptococcus species*), fungi (*Aspergillus oryzae*), yeast (*Pichia species*) and actinomycetes (*Streptomyces coleicor*).

The antibacterial activity of 3a-f compounds was measured on each of these microorganism strains on a potato dextrose agar medium (PDA). Such a PDA medium contained⁵ potato 200 g, dextrose 20 g, agar 30 g, and water 1 Litre. PDA medium autoclaved at 121°C temperature and 15 lbs pressure for 15 minute. After autoclaving the compounds to be tested were inoculated (1000 ppm) in PDA medium at 42 °C temperature and mixed, then these media were poured in to sterile empty glass petriplates. The testing microorganism (young culture) were inoculated after solidification of the PDA medium plates. The percentage inhibition of growth of microorganism was calculated after 5 days of incubation of PDA medium plate inoculated with microorganism at appropriate temperature (Bacteria- 37°C, Fungi- 25°C, Actinomyces- room temp.) percentage of inhibition of microorganism was calculated by using the formula given below.

$$\text{Percentage of inhibition of growth of microorganism} = \frac{100(X-Y)}{X}$$

Where, X= area of bacterial growth in control plate (mm),

Y= area of bacterial growth in test plate (mm).

The antimicrobial activity of all the 3a-f compounds are furnished in Table 2.

Table 2. Antimicrobial activity of 2 (4-chloro phenyloxy) -4,6- bisaryl hydrazino -1,3,5- triazines (3a to f)

Sample	Percentage of inhibition of growth at 1000 ppm concentrate of sample, %						
	<i>Bacillus cereus</i>	<i>Streptococcus species</i>	<i>Escherichia coli</i>	<i>Proteus vulgaris</i>	<i>Aspergillus oryzae</i>	<i>Pichia species</i>	<i>Streptomyces coelicolor</i>
Control	Nil	Nil	Nil	Nil	Nil	Nil	Nil
3-a	78	93	92	91	75	78	65
3-b	98	92	88	93	74	78	62
3-c	83	95	98	87	71	80	79
3-d	81	96	73	62	78	77	75
3-e	95	98	92	80	72	80	78
3-f	85	88	98	72	77	84	89

Results and Discussion

The reaction between aryl hydrazine and 2(4-chloro phenyloxy)-4,6-dichloro-1,3,5-triazine is facile. The products (3a-f) are dark yellow amorphous powders. The C,H,N contents of all (3a-f) shown in Table-1 are consistent with the predicated structures shown in Scheme-1. The IR spectra of all (3a-f) are almost identical. All the IR spectra comprises following important features.

1. -NH-NH- (hydrazine group) : 3280,1610,820 cm^{-1}
2. *s*-triazine : 1510,1250,870 cm^{-1}
3. -NH- (secondary) : 3400 cm^{-1}
4. Aryl-O-Aryl : 1200 cm^{-1}

As the compounds (3a-f) except 3e are insoluble CDCl_3 , the NMR spectral study attempted for 3e. The NMR spectrum of 3e comprises the multiplet between 6.9 to 8.1 δ ppm mainly due to aromatic protons. While the signal at 2.6 δ ppm with integration of 6H is responsible for two CH_3 of OCH_3 groups. The signals in most downfield (9.5 δ ppm) is from NH-NH protons. The result of antimicrobial screening showed (Table 2) that compounds 3a,b,c,f displayed a high order of antibacterial activity and remaining compounds showed weak to moderate activity against both the bacteria. Similarly compounds 3a,d, and f showed higher antifungal activity and remaining compounds displayed moderate antifungal activity against the fungi.

Conclusions

The synthesis of aryl hydrazine-*s*-triazine is facile. The produced compounds have good microbial toxicity. Due to NH-NH groups these compounds can be utilized for epoxy resin hardner. Such work in polymer journal will be published shortly.

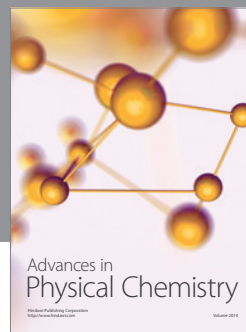
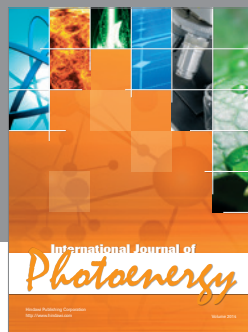
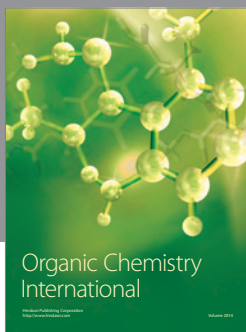
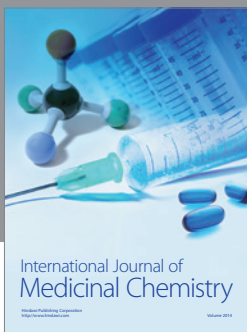
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