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Micellar mediated reactions: Synthesis of substituted phenacyl phenolic ethers

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ABSTRACT

Substituted phenacyl phenolic ethers are synthesised from dihydric phenols with substituted phenacyl bromide in micellar medium. Physical data and biological activities are discussed. These ethers are characterised by C, H, N analysis, IR, ^{1}H NMR, ^{13} NMR data.

Keywords: Dihydric phenolic ether, phenacyl bromide, *Staphylococcus, E-coli.*

INTRODUCTION

Phenacyl ethers have wide range of applications in the modern world. Variety of ethers like, aryl ethers, benzyl ethers, phenacyl ethers, phenyl phenacyl ether, napthayl ethers have been synthesised by different methods and in different environments [1, 2]. The application of phenolic ethers depends upon the nature of the group attached to the other end of the phenolic oxygen. The phenyl ether of ethylene glycol possesses antibacterial properties against *Pseudomonas aerunosa*. The antimicrobial and pharmacological activities of a number of related compounds have been evaluated. The investigation of compounds of this category has shown significant clinical activity. For example, mephenesin, 3-o-toloxy-1, 2-propanediol, is widely used as a muscle relaxant and chlorophenesin, 3-p-chlorophenoxy-1, 2-propanediol, is used in the treatment of fungus infections of the skin [3, 4]. The objective of this investigation is to study the antibacterial and antifungal activity of variety of phenolic ethers newly prepared in this work. Literature search reveals that, diphenyl ethers and their halo derivatives have agrochemical uses such as herbicides and fungicides. Similarly, phenolic ethers are found in pharmaceutical, chemical engineering, food colouring materials, perfumes and additive for polymers [5, 6]. They are also used as intermediates in organic reactions. Owing to their importance, attempts have been made to prepare variety of phenolic ethers and characterise by physical data. Further these compounds have been screened for their biological activity.

MATERIALS AND METHODS

Pyrocatechol, Bromine, Sodium Lauryl sulphate supplied by Merck, substituted acetophenones supplied by Queligens were used as such. Silica gel G. coated TLC plates were used for chromatography. C, H, N analyses were carried out at STIC Cochin University of Science and

Technology, Cochin. IR spectra were recorded on a shimedzu Affinity-1 spectrometer and NMR spectra on Bruker 400M Hz instrument.

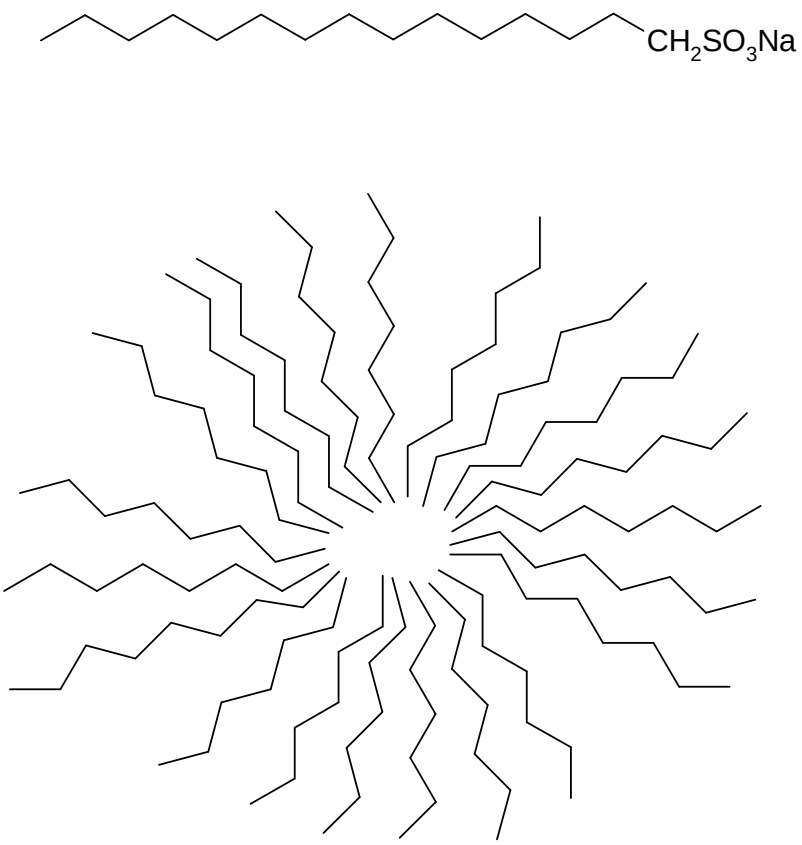
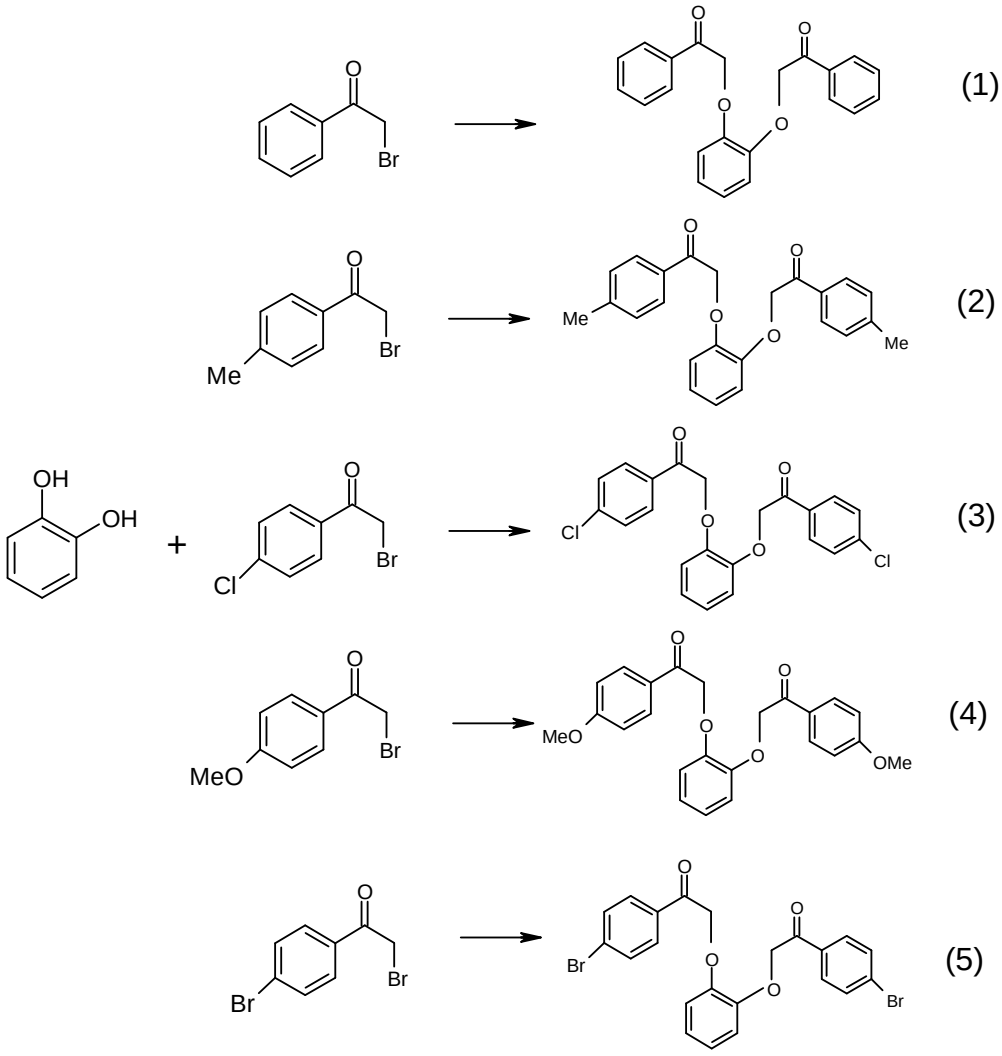


Figure -1 Structure of micelles

Dihydric phenols are treated with methyl, chloro, methoxy or bromo substituted phenacyl bromide in anionic surfactant in the presence of co-surfactant triethylamine [7]. Two moles of substituted phenacyl bromide was dissolved in 25 ml micellar solution and placed in a magnetic stirrer for stirring. One mole of dihydric phenol and two moles of triethylamine was added drop wise to the reaction mixture. The reaction mixture was stirred continuously for 2- 3 hours at room temperature. Overall the reaction was carried out in the micellar solution. Finally, solid product was thrown out, filtered off, washed with water and petroleum ether for several times to remove the unwanted impurities. The product was dried and recrystallized using ethyl acetate solvent. The corresponding product was preliminarily characterised by melting point and TLC. The structure was confirmed by IR, ^{1}H and ^{13}C NMR data.

Scheme-1 is the representative for the formation of the products (1-5) in the case of substrate 1, 2-dihydric phenol. Similar procedure is adopted for 1, 3-dihydric phenol to get the products 6-10. Using 1, 4-dihydric phenol the products 11-15 were obtained.



Scheme.1

RESULTS AND DISCUSSION

The phenacyl phenolic ethers are synthesised in miceller solution. The mechanism of the reaction is that, the alkyl chain of Sodium Lauryl sulphate (NaLS) (Figure -1) has hydrophobic region [8]. The head group sulphate ion of NaLS, possessing negative charge is in the bulk solution which behaves as hydrophilic region. The aromatic part of the reagents such as phenacyl bromide and phenol are highly populated in the micelles, on the other hand, the polar part of the reagents such as $–COCH_2Br$ of phenacyl bromide, -OH of phenol and triethylamine are concentrated in the hydrophilic region. This may facilitate the generation of the phenolate ion to react with substrate to give ether.

Substituted phenolic phenyl ethers are novel compounds because of their significant biological activities. To determine the bacteriostatic and fungistatic activities, micro organisms like pseudomonas aeruginosa, staphylococcus aureus, Escherichia coli, and bacillus subtilis were used in this work. The ether products are characterised by IR (Table.1) and ^{1}H NMR (Table.2), ^{13}C NMR (Table.3). The C, H, N analysis also corroborated the structures assigned.

Table 1 IR spectral data of the compounds (1-15)

Compound no.	C-H Aromatic	C-H Aliphatic	C=O	C=C	C-O-C
1	3108	2940	1696	1524	1226
2	3112	2927	1595	1433	1240
3	3055	2922	1680	1594	1228
4	3057	2918	1683	1502	1216
5	3057	2920	1710	1504	1286
6	3101	2920	1708	1590	1283
7	3056	2910	1686	1489	1228
8	3059	2906	1710	1581	1282
9	3092	2901	1711	1584	1283
10	3054	2925	1678	1493	1228
11	3064	2919	1689	1506	1222
12	3057	2920	1689	1491	1229
13	3082	2956	1696	1585	1226
14	3032	2920	1695	1606	1232
15	3091	2923	1688	1487	1230

Table 2 ^{1}H NMR spectral data the compounds (1-15)

Compound no.	OAr, COAr	-O-CH_2-CO	CH_3
1	7.88-8.03m,6.88-7.63m	5.21s	
2	7.87-7.90m,6.77-7.29m	5.18s	2.37
3	7.83-7.94m,7.18-7.47m	5.14s	
4	7.91-7.99m,6.73-6.97m	5.15s	3.85 *
5	7.74-7.99m,7.19-7.65m	5.52s	
6	7.87-8.02m,7.14-7.63m	5.23s	
7	7.25-7.89m,6.54-7.19m	5.20s	2.42
8	7.45-8.01m,6.46-7.26m	5.18s	
9	7.14-7.99m,6.54-6.97m	5.18s	2.17 *
10	7.15-7.93m,6.47-6.57m	5.18s	
11	7.99-8.03m,6.93-7.72m	5.39s	
12	7.81-7.92m,6.36-7.65m	5.42s	2.24
13	7.39-7.95m,7.01-6.81m	5.32s	
14	7.14-7.99m,6.47-6.97m	5.18s	2.17 *
15	7.46-7.99m,6.78-7.02m	5.34s	

** Data correspond to methoxy methyl group*

Table 3 ^{13}C NMR spectral data of the compounds (1-15)

Compound No.	Ar-C=O	Aromatic Carbon	*Ipso-carbon*	CH_2-O-	CH_3
1	194	134	128	71	
2	194	144	129	71	21
3	193	140	121	71	
4	193	149	130	71	55 *
5	194	134	130	66	
6	194	135	128	70	
7	193	144	130	70	21
8	193	140	129	70	
9	192	130	127	70	55 *
10	193	133	129	70	
11	191	136	128	66	
12	195	148	127	72	21
13	194	147	129	70	
14	192	130	127	70	55 *
15	195	146	131	70	

**Data correspond to methoxy methyl group*

Table 4 Biological data of some phenolic phenacyl ethers

Name of the Organism	Zone of inhibition in mm.								
	Compound 1			Compound 7			Compound 11		
	50 mg	150 mg	250 mg	50 mg	150 mg	250 mg	50 mg	150 mg	250 mg
Staphylococcus aureus (Gram + ve)	11	22	25	12	11	20	9	15	16
Bacillus subtilis (Gram + ve)	10	12	18	11	14	16	12	16	16
Shigella shigal (Gram – ve)	9	15	18	14	17	18	13	15	13
Pseudomonas aruginosa (Gram – ve)	12	10	15	12	15	15	10	18	20

Table 5 Biological activity data of the compounds (1-10)

Compound no	Zone of inhibition	Staphylococcus	E-coli
1	50 μg	10	10
	150 μg	16	15
2	50 μg	11	12
	150 μg	14	14
3	50 μg	13	18
	150 μg	18	20
4	50 μg	14	10
	150 μg	16	16
5	50 μg	16	10
	150 μg	21	12
6	50 μg	10	12
	150 μg	13	14
7	50 μg	10	10
	150 μg	10	13
8	50 μg	22	11
	150 μg	14	14
9	50 μg	12	12
	150 μg	14	16
10	50 μg	12	12
	150 μg	18	18
	Std	30	38

Standard (Std)-ciprofloxacin 5 μg /disc for bacteria
Solvent control (Sc) -DMSO

CONCLUSION

In vision of the significance of these ether compounds, it is planned to syntheses various new phenolic phenacyl ethers from dihydric phenol with substituted phenacyl bromide. It is intended to know the physical and spectral data of these compounds to assign their structures. In this work it is used to take electron withdrawing and electron donating group as the substituent in phenacyl bromide which reacts with dihydric phenols and gives different type of phenolic phenacyl ethers. IR, ^{1}H NMR, ^{13}NMR are used to identify the new compounds. Anti microbial and bacterial activities were carried out for phenolic phenacyl ethers

REFERENCES

[1] Moroz AA, Shaurtsbert Russian, *Chemical Reviews*, **1974**, 679.

[2] Robert B Bates, Kim D Janda, *J.Org.Chem*., **1982**,47,4374,
[3] Berge FM, Hubbard CV , Ludwig B J, *Applied microbial*, **1953**, 3, 146.
[4] Berger FM , Bradley W, *Brit, J.Pharmacol*., **1946** ,1, 265.
[5] Hartley F, *Quart. J.Pharm. and Pharmacol*., **1947**,20, 388.
[6] Fritz K roenke, Gertrauae Gern, Wand Ahrenholz, *J.Prakt. Chem*., **1960**,11, 239.
[7] Rico I, Halvorsen K, Dubrule C, Lattes A, *J.Org.Chem*., **1994**,59,415,
[8] Fendler JH, Fendler JH, *Catalysis in micellar and macromolecular System*, Academic press, New York, **1975**.