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One pot synthesis of Dibenzoxanthenes using *p*-Toluene Sulphonic Acid under Solvent Free Condition

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Abstract –A one pot condensation reaction of aromatic aldehydes and beta-naphthol has been described using p-toluene sulphonic acid as a catalyst under solvent free condition. Short reaction time, higher yields and clean reaction makes this protocol an attractive alternative to the existing methods.

Keywords: aldehydes, beta naphthol, solvent free

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Introduction:

Xanthenes and its derivatives are known as an important class of heterocyclic compounds widely used as leco-dye in laser technology and pH sensitive flurescent materials .Although not widely found in nature,xanthenes and compounds based on these core templates exhibit a broad spectrum of pharmaceutical activities.[1-4]

These compounds are also utilized as antagonist for paralyzing action of zoxazolamine and in photodynamic therapy.[5]Thus a broad utility range has made xanthenes prime synthetic candidates thereby accentuating the need to develop newer synthetic routes. Xanthenes and benzoxanthenes constitute important classes of biodynamic heterocycles and their synthesis has received much attention especially in the field of medicinal/pharmaceutical chemistry due to their wide range of biological/pharmacological activities, e.g., anti-inflammatory,[4] antiviral[6] and antibacterial.[7]Some of them have been used as antagonists for paralyzing the action of zoxazolamine[8] and in photodynamic therapy.[9] In addition,their derivatives can be used as dyes,[10]pH sensitive fluorescent materials for the visualization of biomolecular assemblies[11] and in laser technologies[12]for scaffold manipulation of xanthene derivatives. The dibenzoxanthenes have been prepared by nTSA,[13] using trichloroacetic acid as heterogeneous catalyst under solvent free conditions.[14]

The increasing demand for clean and efficient chemical reactions result in solvent free reaction condition which is of great current interest. Also, economic and environmental concern encourages the applications of heterogeneous catalyst to carry out various organic transformations. This catalyst can conveniently be handled and removed from the reaction

mixture, making the experimental procedure, ecofriendly and simple. Therefore performing an organic reaction using a simple and efficient catalyst would be an ideal methodology, if the catalyst shows high catalytic activity under solvent free condition.

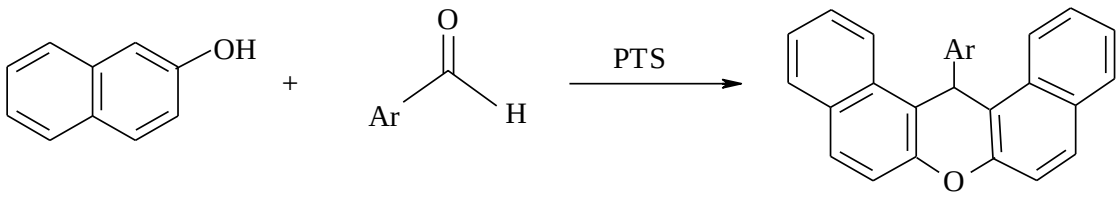
The synthesis of xanthenes and their derivatives has received significant attention due to their pharmacological activities. Xanthenes molecule have wide spectrum of activities like antibacterial, anti-inflammatory, antiviral and anticancer etc. These compounds have also been employed as sensitizers in photodynamic therapy in the food industry as additive in LASER technology as fluorescent materials for the visualization of biomolecules. In addition, xanthenes and their derivatives can be used as sensitizer in dye sensitizer solar cells. Xanthenes are rare in natural plants and have been isolated from only two plant families, *Compositae* and *Fabaceae*

Experimental:

Typical experimental procedure for the synthesis of Dibenzoxanthenes :

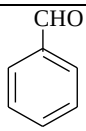
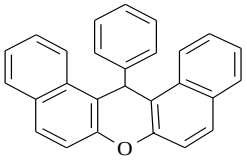
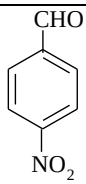
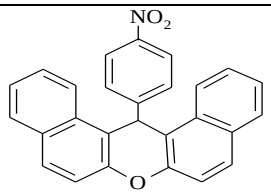
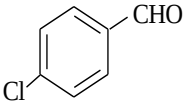
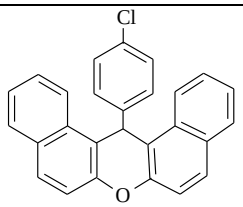
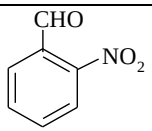
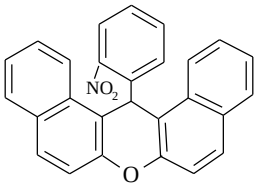
A mixture of an aldehyde (10 mmol), 2-naphthol (20 mmol) and *p* -toluene sulphonic acid (0.5 g) was stirred at 120^0c for the appropriate time indicated in table. The progress of reaction was monitored by TLC (ethyl acetate/n-hexane). After completion of the reaction, the reaction mixture was cooled at room temperature and water (100 ml) was added and the mixture was stirred for 10 min. The obtained solid was collected by filtration and purified by recrystallization from ethanol.

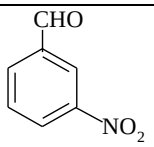
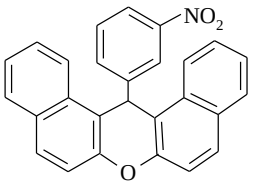
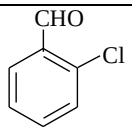
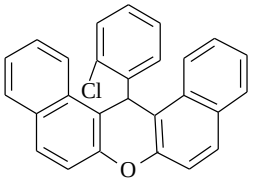
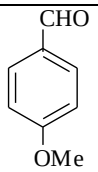
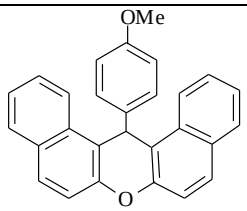
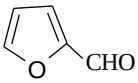
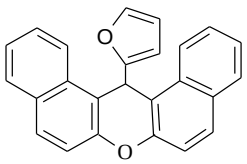
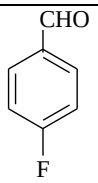
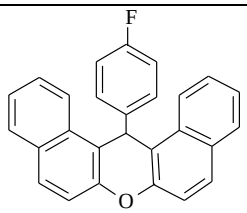
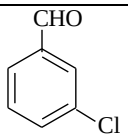
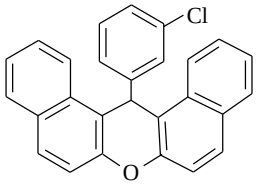
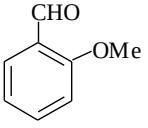
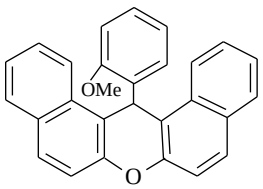
Scheme:



1 **2** **3a-k**

Characterization Table:

Entry	Aldehyde	Product	Time min.	Yield (%)	M. p.°C [Lit.][13,14]
1		 **3a**	15	76	188-190 [190-193]
2		 **3b**	10	82	326-328 [325-326]
3		 **3c**	10	84	295-296 [295-298]
4		 **3d**	10	93	215-216 [213-214]

5		 **3e**	10	90	216-218 [211-215]
6		 **3f**	05	93	218-220 [216-218]
7		 **3g**	15	83	210-212 [209-210]
8		 **3h**	15	79	189-191
9		 **3i**	10	87	238-240 [238-239]
10		 **3j**	10	84	246-248
11		 **3k**	15	76	268-270 [264-268]

Spectral Analysis:-

IR data:

Compound:(**3a**)**:** υcm$^{-1:}$1255 (C-O),1619(C=C), 3061(C-H), 739(o-disubustituted) .

Compound:(**3c**)**:** υ cm^{-1} : 802(C-Cl), 1033(C-O), 1619(C=C), 3050 (C-H), 739 (monosubstituted).

Conclusion:

In conclusion, we report herein one-pot synthesis of dibenzoxanthenes in the presence of p-toluene sulphonic acid under solvent free condition at 120^{0}C. This method offers some advantages in terms of solvent free condition, low cost and it follows along the line of green chemistry. The catalysts is readily available and inexpensive and conveniently be handled and removed from the reaction mixture. In many cases, the products crystallized directly from the reaction mixture in high purity.

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