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Synthesis of xanthene using chloro sulphonic acid as a efficient catalyst

R. B. Kshatriya[1] and S. T. Salunke[2]*

[1]Post Graduate Department of Chemistry, Poona College of ACS, Camp, Pune
[2]Department of Chemistry T.C. College, Baramati (Affiliated to University of Pune), Maharashtra, India

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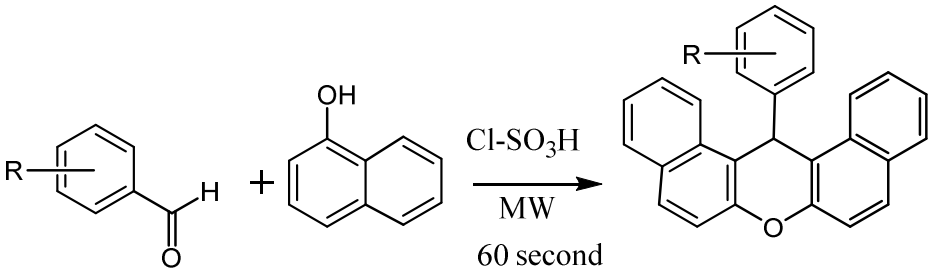
ABSTRACT

Multicomponent reactions plays important role in the synthesis organic compounds. Products are obtained in one pot hence cost of production is very low. Another important thing impurity free product obtained. We were synthesized xanthene's using chlorosulphonic acid as a catalyst by one pot method in short span of time.

Keywords: Xanthene, chlorosulphonic acid, multicomponent reaction.

INTRODUCTION

Multicomponent reactions are very important in drug discovery to reduce number of steps .More than two components added in pot and product obtained in single step is beauty of this reaction.Xanthene molecules have wide spectrum of activities like antibacterial[1], anti-inflammatory[2], antiviral[3] & anticancer[4] etc. Various reagents like sulphamic acid[5] ,InCl[6], pTSA[7],CsF[8],$MeSO_3H$[9],$FeCl_3$[10], $ClSO_3H$[11],DABCO[12,13],iodine[4] etc. are used in the synthesis of xanthenes.Microwave technique is important tool for the organic synthesis and preferred over thermal synthesis.Here we applied mw technique.



Scheme1. : Synthesis of xanthene using one pot method

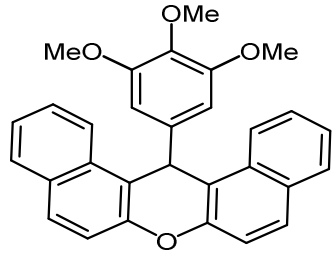
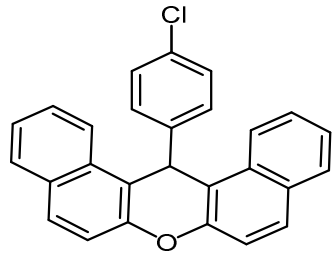
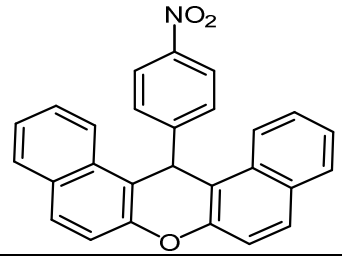
MATERIALS AND METHODS

All the organic chemicals and solvents were obtained from commercial sources (Sd fine, Loba) and used without further purification. Melting point was taken on a on paraffin oil using thiel's tube. Structure of compounds conformed by IR, NMR spectral data.

Representative Procedure for synthesis of xanthenes

In the present work, a mixture of Para-nitro benzaldehyde (1 mmol), β-naphthol (2 mmol) and Chlorosulphonic acid (0.02 mmol) as a catalyst in a RBF fitted with condensor was exposed to microwave irradiation for 60 second. The reaction mixture was homogenized with the help of glass road and the reaction was completed within two successive irradiation. The reactions were monitored by TLC. After completion of the reaction, crude reaction mixture was poured on crushed ice. Solid obtained was filtered, dried, recrystallized from ethanol.M.P.and yield was recorded

Table 1. Xanthene molecules synthesized by one pot method.

Entry	Molecule	Yield	Melting point	
			Found	Reported
A		80	300	Novel
B		89	287	289
C		76	306	310

RESULTS AND DISCUSSION

The objective of present research work is to provide simple and efficient methodologies for the synthesis of Aryl-14 H- dibenzo (a) xanthenes derivatives from aromatic aldehyde and 2-naphthol in presence of chlorosulphonic acid as a catalyst under solvent free conditions. The reaction proceeded smoothly under microwave assisted solvent free condition to offer excellent yields. Initially, we carried out the reaction of 2-naphthol and 4-nitro benzaldehyde in the presence of chlorosulphonic acid catalyst under microwave irradiation using different reaction condition. Excellent yield results were found when the reaction was carried out neat. Similarly the mole ratio of chlorosulphonic acid was studied. It was found that the amount of $ClSO_3H$ affects the yield of the desired product, with 0.02 m mole of $ClSO_3H$ being optimum (**Table 1**).

Spectral Data

14[4- Chlorophenyl]-14 H dibenzo xanthene.(a)

White solid M.P. 287-289^{0}C (lit. Value 289^{o}C)]; **IR [KBR] V_{max} cm^{-1}** 2925, 1590, 1484.; **^{1}H NMR [300 MHz DMSO]** ∂ 7.33 (d,2H,J= 7.2Hz),7.34(d,2H,J=7.4 Hz) 6.80(s,1H) 6.96(dd,2H,J= 7.5,1.5 Hz),7.27(d,2H,J= 7.5 Hz),7.59(dd,2H,J= 1.5,7.0Hz),7.86(d,2H,J = 7.5 Hz),7.82(d,2H,J = 7.5 Hz),6.99(d,2H,J = 7.5 Hz).

14[4- nitro phenyl]-14 H dibenzo xanthene. (b)

Yellow solid M.P. 306-308 (lit. Value 310^{o}C) ; **IR [KBR] V_{max} cm^{-1}** 2930, 1594, 1517. ; **^{1}HNMR[300 MHz DMSO]** ∂ 7.45(d,2h,j=7.9 Hz),8.56(d,2H,J=7.5 Hz),6.80 (s,1H),6.96(dd,2H,J= 7.5,1.5 Hz),7.27(d,2H,J= 7.5 Hz),7.59(dd,2H,J= 1.5,7.0Hz),7.86(d,2H,J = 7.5 Hz),7.82(d,2H,J = 7.5 Hz),6.99(d,2H,J = 7.5 Hz).

14-(3, 4,5-trimethoxyphenyl)-14H-dibenzo[a,j]xanthene (c)

Orange colour**,** M.P.300^{0}C; **IR [KBR] V_{max} cm^{-1}** 2900, 1584, 1500.; **^{1}H NMR [300 MHz DMSO]**∂ 6.50(d,2H,J= 1.5 Hz),3.72(9H,S,3O-Me),5.7(s,1H),6.96(dd,2H,J= 7.5,1.5 Hz),7.27(d,2H,J= 7.5 Hz),7.59(dd,2H,J= 1.5,7.0Hz),7.86(d,2H,J = 7.5 Hz),7.82(d,2H,J = 7.5 Hz),6.99(d,2H,J = 7.5 Hz).

CONCLUSION

In this present project we are reporting the most convenient way to synthesize the Xanthene derivatives in which microwave irradiation plays an important role for promoting condensation reaction of P-nitrobenzaldehyde and 2-naphthol. In conclusion, in this work we investigated a simple efficient and rapid method for the synthesis of aryl 14H-dibenzo [a, j] xanthenes under microwave irradiation method.

The application of microwave irradiation in combinatorial chemistry becomes potent device in accelerating the pace of library synthesis. Domestic microwave oven is very popularity used in organic synthesis because of its low cost and easily available, shorter reaction time and products are obtained in excellent yields. Furthermore this methodology also follows several principles of green chemistry.

Acknowledgments

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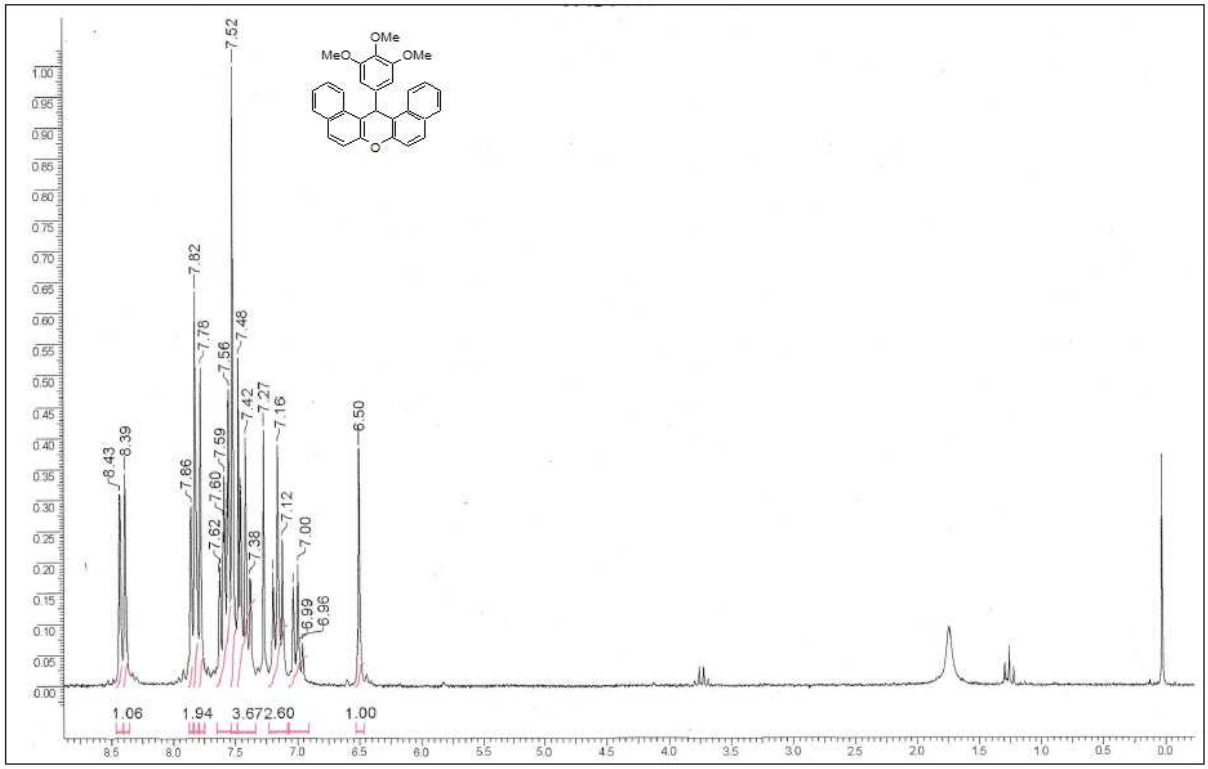
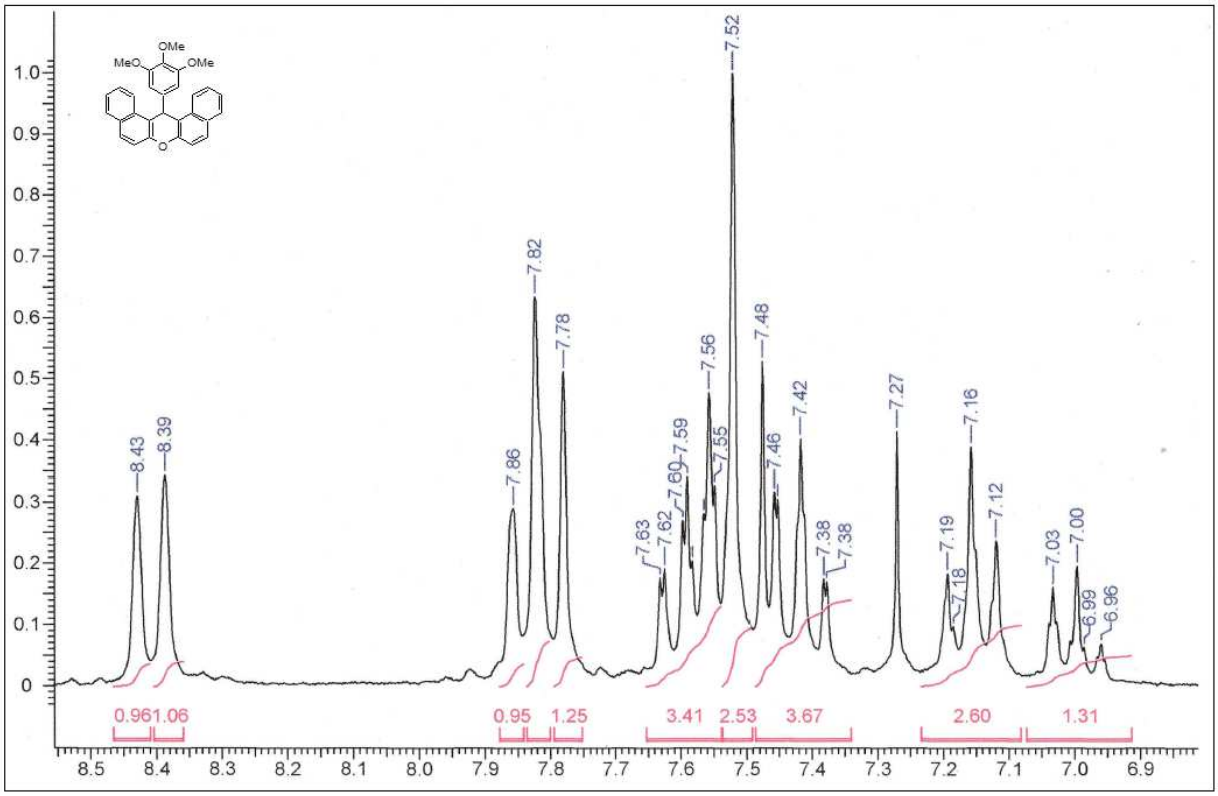
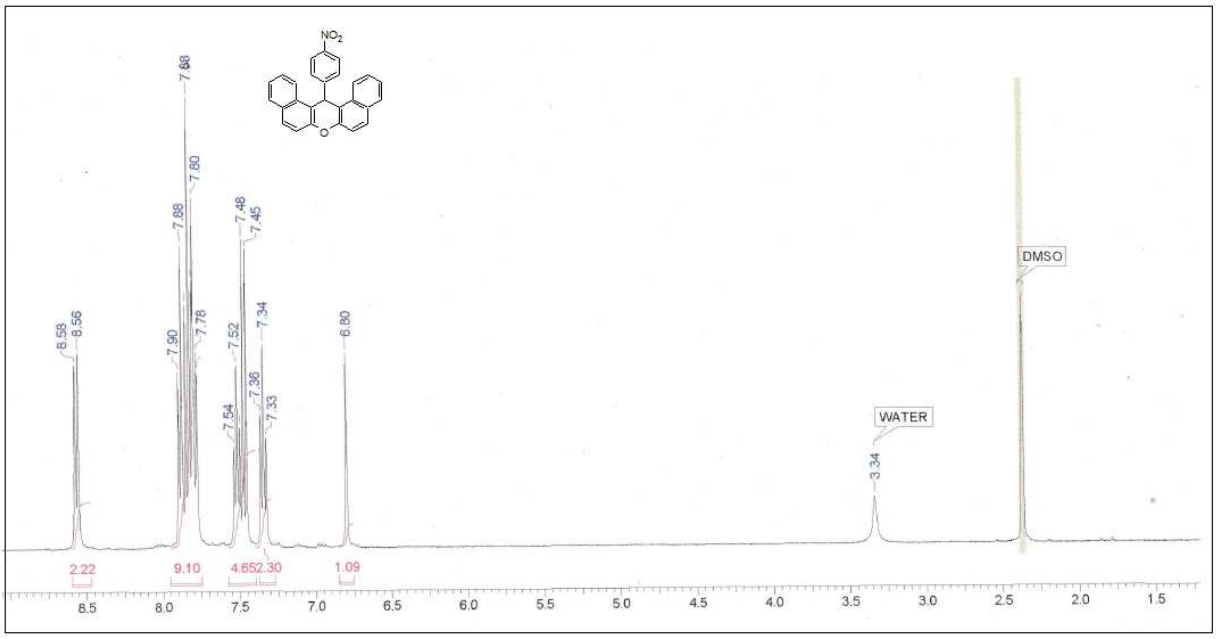
Supplementary data.
Compound 1.





Compound 2.



Compound 3.