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# An efficient and improved method in the synthesis of 14-alkyl and 14-aryl-14H-dibenzo [a,j]xanthenes using 1-butyl-3-methyl imidazolium phosphotungstate as catalyst under solvent free conditions

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**Abstract.** 1-Butyl-3-methyl imidazolium phosphotungstate was used successfully as a mild catalyst in the synthesis of 14-ethyl and various 14-aryl-14H-dibenzo [a,j]xanthenes derivatives via condensation reaction of acetaldehyde and different aromatic aldehydes with 2-naphtol. This study represents a simple, efficient, and easily recoverable catalyst in the synthesis of biologically active molecules. The project included the search for the optimum composition of the hybrid catalyst and the study of reusability of prepared catalyst.

## 1. Introduction

In recent years, heteropolyacids (HPAs) have attracted more attention than ever before. The main reason is attributed to the tenability of their chemical properties [1]. The day to day growing usage of HPAs as anti-cancer drugs [2], fuel cell membranes [3,4], thiophenehydrodesulfurization reagents [5], $CO_2$ sorption compounds [6], and catalysts in chemical reactions [7-13] has brought them fully to the attention of chemists and biochemists.

The great thermal stability of heteropolyacids has, so far, made these compound great catalysts for high temperature reactions. However, difficult recovery of the catalyst, as well as the need for toxic solvents during the process, makes these catalysts less environmental friendly. On the other hand, supported heteropolyacids show more catalytic reactivity due to their high surface area, convenient catalyst recovery, and higher catalyst turn over [14].

At present, organic-inorganic hybrid materials, which consist of inorganic polyoxymetalate and ionic liquids, have gradually gained broader attention compared to simple polyoxymetalates. These heterogenous catalysts combine the benefits of ionic liquids, such as simple and easy recovery, to further wide applications of HPAs [15-18].

In this work, $[BMIM]_3[PW_{12}O_{40}]$ was synthesized through the known procedure by Rajkumar, as shown in Scheme 1 [19]. The catalyst was successfully used in the synthesis of 14-alkyl and 14-aryl-14H-dibenzo [a,j]xanthenes (Scheme 2).

Xanthene derivatives are well known for their biological and therapeutic properties, such as anti-viral [20], anti-bacterial [21], and anti-inflammatory [22]. Moreover, they have been used

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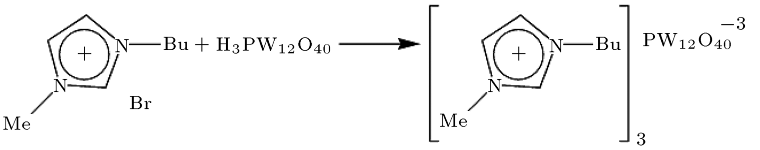
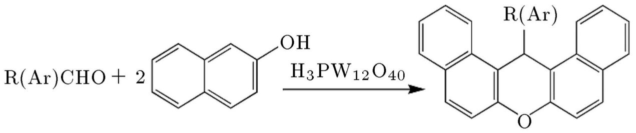


**Scheme 1.** Synthesis of 1-butyl-3-methyl imidazolium phosphotungstate.



**Scheme 2.** Condensation reaction of aldehydes and 2-naphtol.

in dye industries [23], laser technologies [24], and fluorescent materials [25].

## 2. Experimental

### 2.1. *General*

IR spectra were obtained with neat thin films on a potassium bromide disc, and were recorded on a BOMEM MB-Series 1998 spectrophotometer (FT-IR). $^1$H NMR spectra were recorded on a Bruker DPX 400 (400 MHz) spectrometer at ambient temperature. The progress of the reaction was followed with Thin-Layer Chromatography (TLC) using silica gel SILG/UV$^{254}$ plates. All products are known and were characterized via a comparison of IR, $^1$H NMR, $^{13}$C NMR obtained spectral data and melting points with their reported literature values.

### 2.2. *Synthesis of 1-butyl 3-methyl imidazolium phosphotungstate [19]*

1-butyl 3-methyl imidazolium bromide (3 g, 0.0137 mol) was added drop-wise to the solution containing 14.3 g (0.005 mol) of phosphotungstic acid (PWA) under constant stirring at room temperature. The obtained white precipitate was washed with distilled water until it was free of bromine (checked with $AgNO_3$), and dried overnight at 80°C.

### 2.3. *General procedure for the preparation of 14-aryl-14H-dibenzo [a,j]xanthene*

A mixture of $[BMIM]_3$ $[PW_{12}O_{40}]$ (0.5 g), aldehyde (0.5 mmol), and 2-naphthol (1 mmol) was stirred at 120°C. Reactions were monitored by TLC. After about 1 hour, the reactions were complete. Hot ethanol was added to the mixture and the catalyst was filtered off. The pure products were recrystallized by means of ethanol. Isolated yields of products were 81-97%.

Spectral data for 14-(4-chlorophenyl)-14H-dibenzo [a,j]xanthene: M.p 289-291°C; IR (KBr) ($\nu_{\max}$,cm$^{-1}$): 3040, 1625, 1582; $^1$H NMR ($CDCl_3$) $\delta_H$6.44 (1H, s, CH), 7.10-8.34 (16H, m, Arom.); $^{13}$C NMR ($CDCl_3$): $\delta_C$ 37.4, 116.8, 118.2, 122.5, 124.37, 126.9, 128.7, 128.9, 129.1, 129.5, 131.1, 131.3, 132.0, 143.5, 148.7.

**Table 1.** The effect of different amounts of catalyst and temperature on the synthesis of 14-phenyl-14H-dibenzo[a,j]xanthenes.

| **Entry** | **Cat. (g)** | **Temp. (°C)** | **Time (h)** | **Yield (%)** |
|---|---|---|---|---|
| 1 | 0.161 | 120 | 4/5 | 42 |
| 2 | 0.25 | 120 | 3/5 | 50 |
| 3 | 0.4 | 120 | 3 | 72 |
| 4 | 0.5 | 120 | 2 | 92 |
| 5 | 0.8 | 120 | 3 | 75 |
| 6 | 0.5 | 90 | 5 | 60 |
| 7 | 0.5 | 100 | 5 | 66 |
| 8 | 0.5 | 110 | 4 | 75 |
| 9 | 0.5 | 130 | 2 | 90 |

## 3. Results and discussion

In the present work, the catalytic activity of $[BMIM]_3$ $[PW_{12}O_{40}]$ was tested in a model system using benzaldehyde and 2-naphthol (Scheme 2) under solvent-free conditions. In the absence of a catalyst, no reaction was observed at 80°C and only traces were obtained at 120°C for a reaction time of about 1 h. The optimized conditions were determined by varying the time, temperature and number of the equivalents of each reagent.

As shown in Table 1, the best result was obtained by carrying out the reaction using $[BMIM]_3$ $[PW_{12}O_{40}]$ (0.5 g), 2-naphthol (1 mmol), benzaldehyde (0.5 mmol), at 120°C (Table 1, entry 4). Changing the amount of the catalyst and the temperature of the reaction did not improve the yields and reaction times. In addition, results indicated that when the reaction proceeded at room temperature for 5 h, the yield of the corresponding product was low.

Other control experiments were carried out to clarify the role of the reactants. The generality of the method was demonstrated using different types of aldehydes containing either electron donating or electron withdrawing groups. All reactions proceeded rapidly, and complete conversions were observed in less than 5 hours (Table 2). Aldehydes with electron withdrawing groups showed slightly higher conversion in shorter reaction time compared to aldehydes with

**Table 2.** Three component one-pot synthesis of 14-ethyl and 14-aryl-14H-dibenzo[a,j]xanthenes.

| Entry | Aldehyde | Time | M.p. (°C) | | Yield[a] (%) |
|---|---|---|---|---|---|
| | | | Found | Reported | |
| 1 | $C_6H_5CHO$ | 1h | 182-184 | 182-183 [26] | 90 |
| 2 | $p$-$ClC_6H_4CHO$ | 4 h | 289-291 | 290-291 [26] | 92 |
| 3 | $o$-$ClC_6H_4CHO$ | 4 h | 213-215 | 213-214 [26] | 91 |
| 4 | $m$-$ClC_6H_4CHO$ | 3 h | 208-210 | 209-210 [27] | 90 |
| 5 | $p$-$NO_2C_6H_4CHO$ | 75 min | 310-311 | 312-313 [26] | 97 |
| 6 | $o$-$NO_2C_6H_4CHO$ | 100 min | 213-215 | 213-215 [28] | 93 |
| 7 | $m$-$NO_2C_6H_4CHO$ | 110 min | 212-214 | 210-211 [27] | 94 |
| 8 | $p$-$MeC_6H_4CHO$ | 5 h | 227-228 | 226-228 [26] | 85 |
| 9 | $p$-$MeOC_6H_4CHO$ | 5 h | 204-205 | 204-205 [26] | 83 |
| 10 | $m$-$MeOC_6H_4CHO$ | 5 h | 176-178 | 175-177 [27] | 82 |
| 11 | EtCHO | 3 h | 150-151 | 150-151 [29] | 81 |

[a]: Yields refer to isolated and pure products.

**Table 3.** Comparison of the results obtained by the present method with some of the previous methods for 14-alkyl and 14-aryl-14H-dibenzo[a,j]xanthenes.

| Entry | Catalyst | Temperature (°C) | Time (min) | Yield[a] (%) |
|---|---|---|---|---|
| 1 | 1-butyl-3-methyl imidazolium phosphotungstate | 120 | 1 h | 90 |
| 2 | Phosphosulfonic acid | 110 | 35 | 93 [30] |
| 3 | Acyclic Br$\phi$nsted acidic ionic liquid [NMP][$HSO_4$][b] | 110 | 12 | 94 [31] |
| 4 | Highly sulfonated single-walled carbon nanotube | 70 | 8 | 93 [26] |
| 5 | Iron (III) triflate | 60 | 4 h | 88 [32] |
| 6 | $Fe_3O_4$ nanoparticles | 100 | 30 | 88 [33] |
| 7 | $CuSO_4.5H_2O$ | 80 | 5 h | 95 [34] |
| 8 | Polyvinylpolypyrrolidone-supported boron trifluoride | 120 | 30 | 94 [35] |
| 9 | Nanoporous $Na^+$-montmorillonite sulfonic acid | 120 | 10 | 91 [36] |
| 10 | N-sulfonic acid poly(4-vinylpyridinium) chloride | 100 | 30 | 80 [37] |

[a]: Yields refer to isolated and pure products;

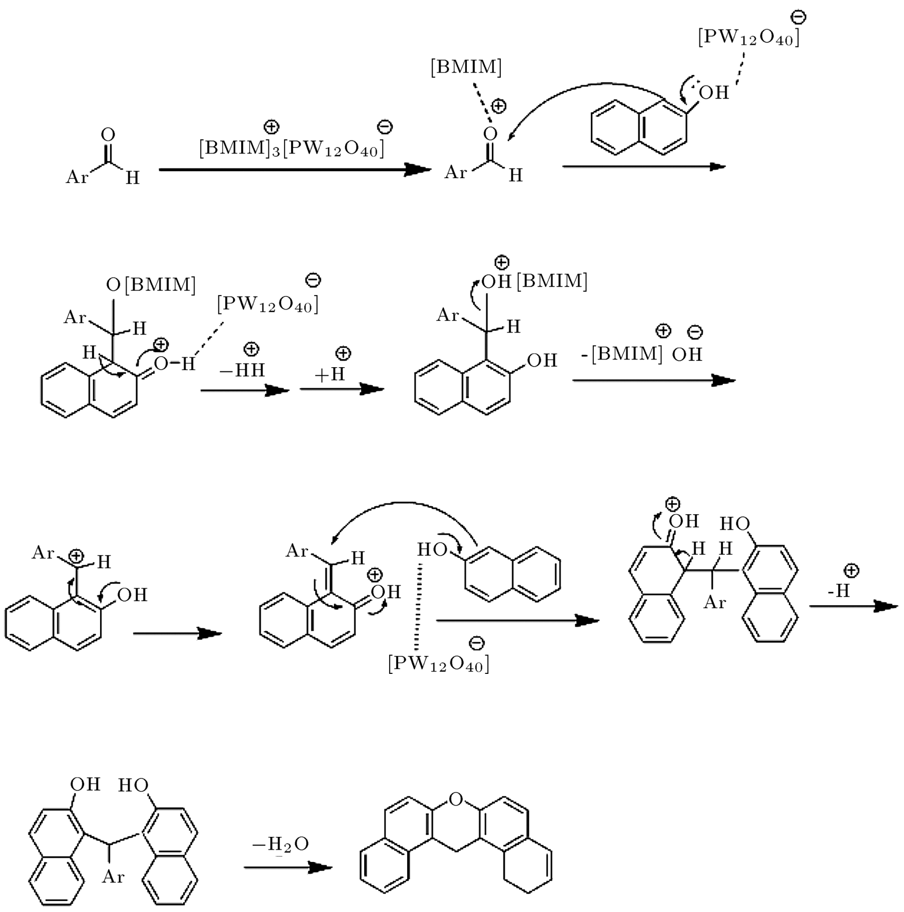
[b]: N-methyl-2-pyrrolidone hydrogensulfate.

electron donating groups (Table 2, entries 2-10). Apparently, the steric bulk of ortho-substituted aromatic aldehydes did not have any effect on the yield of the titled compounds (Table 2, entries 3 and 6). Applying aliphatic aldehydes under the same reaction conditions provided reasonable yield in 3 h (Table 2, entry 11) [26-29].

In order to show the merit of the present work in comparison with recently reported protocols, we compared the time, temperature and yield of our method for the synthesis of 14-alkyl and 14-aryl-14H-dibenzo[a,j]xanthenes with the literature (Table 3) [30-37].

However, some of these methods suffer from certain drawbacks of green chemistry principles. These include long reaction times, low yields of products, use of organic solvents, and the use of toxic, corrosive and expensive catalysts. Moreover, the reusability of the catalyst is a problem. Therefore, the introduction of a clean procedure utilizing a green, efficient and more economic catalyst that can be easily recycled is needed for the production of 14-alkyl and 14-aryl-14H-dibenzo [a,j]xanthenes.

The reusability of the present catalyst was studied, so that after each run, hot ethanol was added to the mixture and the catalyst was filtered off. To recycle the catalyst, the collected catalyst was washed with hot ethanol (3 × 10 ml) to remove organic impurities. Then, the catalyst was dried. It was found that no substantial loss of activity was observed up to six



**Scheme 3.** Proposed mechanism for 14-aryl-14H-dibenzo [a,j]xanthenes synthesis with $[BMIM]_3$ $[PW_{12}O_{40}]$.

cycles. It was also observed from the IR spectral studies that there was no change in the nature of the catalyst, even after six cycles.

Finally, it is observed that the present catalyst has the advantage of easy preparation, mild reaction condition, and simple recovery and reusability. It also does not require any special handling and storage precautions in comparison with other strong acidic catalysts. A plausible mechanism has been offered for the synthesis of 14-aryl-14H-dibenzo[a,j]xanthenes derivatives in the presence of $[BMIM]_3$ $[PW_{12}O_{40}]$ under solvent-free conditions (Scheme 3).

## 4. Conclusion

In summary, the efficient one-pot synthesis of 14-alkyl- or aryl-14H-dibenzo [a,j]xanthenes by a condensation reaction of 2-naphthol with aliphatic or aromatic aldehydes, catalyzed by 1-butyl-3-methyl imidazoliumphosphotungstate, has been demonstrated. The present protocol illustrates the use of a simple, high yielding, and easily recoverable catalyst in the synthesis of a diverse family of xanthene derivatives. We believe that the present protocol may be a reasonable alternative to existing processes.

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