Asian Journal of Chemistry; Vol. 30, No. 10 (2018), 2343-2346

ASIAN JOURNAL OF CHEMISTRY

https://doi.org/10.14233/ajchem.2018.21498

Synthesis of Xanthenediones by Silica Supported Orthophosphoric Acid (H_3PO_4·SiO_2)

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Received: 5 June 2018; *Accepted*: 16 July 2018; *Published online*: 31 August 2018; AJC-19071

The novel protocol was developed for the synthesis of xanthenediones by silica supported orthophosphoric acid (H_3PO_4·SiO_2) as a heterogeneous catalyst. The reported protocol is simple, scalable, mild and effective for the synthesis of xanthenediones. The (H_3PO_4·SiO_2) catalyst demonstrated excellent catalytic activity for various substituted aromatic aldehydes. This catalyst can be reused four times without much loss in catalytic activity.

Keywords: Xanthenediones, Heterogeneous catalysis, H_3PO_4·SiO_2, Condensation reaction.

INTRODUCTION

In recent years the xanthene fascinated considerable interest to organic synthesis since it exhibits antibacterial, antiviral, anticoagulant, anticancer, diuretic, spasmolytic and anti-inflammatory properties [1,2]. Besides, these compounds have been explored for agricultural bactericidal activity and photodynamic therapy [3,4]. Xanthenediones are integral part of number of natural products [5]. The presence of pyran ring in xanthenediones makes it as versatile synthons [6]. Moreover, their applications are explored in cosmetics, pigments, laser technologies [7,8] and in fluorescent material for revelation of biomolecules [9,10].

In synthesis of xanthenediones, usually acid or base catalyzed condensation of suitable active methylene group containing carbonyl compounds with aldehydes is carried out [11]. In literature different methods have been reported for synthesis of xanthenediones such as condensation of active methylene compounds with aldehydes catalyzed by sulfuric acid or hydrochloric acid [12], TiO_2/SO_4^{2-} [13], polyaniline *p*-toluenesulfonate [14], PPA-SiO_2 [15], Amberlyst-15 [16], Fe^{3+}-montmorillonite [17], $NaHSO_4$-SiO_2 or silica chloride [18], cellulose-sulfuric acid [19], $InCl_3$/ionic liquid [20], Dowex-50W [21], $ZrOCl_2 \cdot 8H_2O_4$, trimethylsilyl chloride (TMSCl) [22], ω-4°-ammoniumalkyl sulfonate [23], polytungstozincate acid [24] and cyanuric chloride [10]. However these methods require harsh reaction conditions, long reaction time, low yields, use toxic and expensive catalysts. So in preparation of xanthenediones, novel methods are desirable, which overcomes afore mentioned drawbacks.

Heterogeneous catalysis has more advantages than homogeneous catalysis because in industrial practice, the removal of the product and recovery of the catalysts are comparatively easier. The development of nonmetallic heterogeneous catalyst is essential due to their advantage of nominal product contamination from metal release during reaction. Recently, silica supported catalysts such as $HClO_4/SiO_2$ [25], H_3PO_4·SiO_2 [26] and ionic liquid prompted microwave irradiation [27] have been used by various research group for organic transformation.

Herein, we report silica supported orthophosphoric acid as a novel heterogeneous, reusable catalyst for synthesis of xanthenediones. To the best of our knowledge, catalyst (H_3PO_4·SiO_2) has not been reported earlier for synthesis of xanthenediones (**Scheme-I**). This catalyst was found to be highly efficient, recyclable and environmentally benign for synthesis of xanthenediones.

EXPERIMENTAL

All commercially available reagents were used as received without further purification. ^1H NMR spectra were recorded