Simple and efficient alkylation of 1,3-dicarbonyl compounds and synthesis of 4*H*-chromenes using $NaHSO_4/SiO_2$

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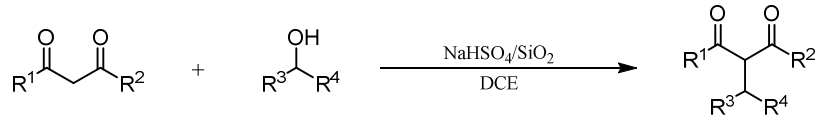
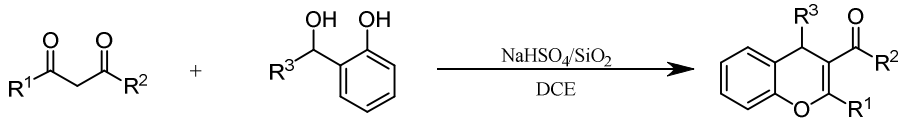
We have developed a simple and efficient procedure for the C-C bond formation between alcohols and active methylene containing compounds using silica-gel supported sodium hydrogen sulfate ($NaHSO_4/SiO_2$) under mild conditions. This method was applied to the synthesis of chromenes. $NaHSO_4/SiO_2$ can be reused for the alkylation without loss of catalytic activity at least 10 times.

Reaction of **1** with **2** was carried out in the presence of $NaHSO_4/SiO_2$ at 80 °C in dichloroethane (DCE) to give corresponding **3** in moderate to excellent yields; eg. acetyl acetone **1a** (R^1, R^2 = CH_3) and benzhydrol **2a** (R^3, R^4 = Ph) were reacted for 30 min to give **3a** in 98 % yield.



1 **2** **3**

When *o*-hydroxy benzhydrol **4** was used in place of **2a**, consecutive alkylation and intramolecular cyclization occurred to give 1-(2-methyl-4-phenyl-4*H*-chromen-3-yl)ethanone **5** quantitatively. Similar reactions using *o*-hydroxy benzylic alcohols and 1,3-dicarbonyl compounds gave the corresponding chromens in moderate to good yields.



1 **4** **5**

Some text