IJETIR

ISSN (Online) 2583-0554

International Journal of Emerging Technologies and Innovative Research (IJETIR)

Volume 3, Issue 3, March 2023

Impact Factor: 5.731

Efficient Synthesis of One- Pot Knoevengel Condensation with Potassium Carbonate in PEG

Nitin Shivaji Pawar, Mhasalkar Vishakha Dipak and Khatib Shifa Maqsood

Department of Chemistry

Anjuman Islam Janjira Degree College of Science, Murud-Janjira, Maharashtra, India

Abstract: *The Knoevengel condensation of aldehydes with active methylene compound proceeded efficiently at room temperature with potassium carbonate as a catalyst and PEG as a green solvent. C-C bonds formation reactions are important derivation in perfume, pharmaceutical, polymer applications.*

Keywords: Knoevengel condensation, Potassium Carbonate, Green Chemistry, PEG, Room Temperature

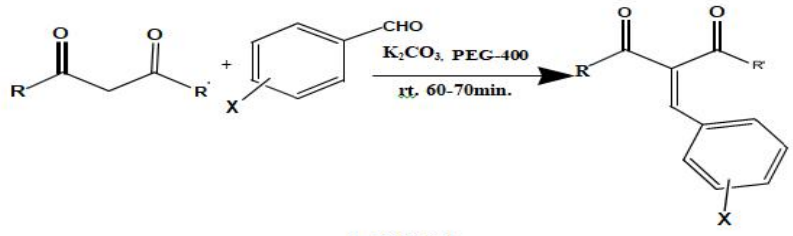
I. INTRODUCTION

The development of cleaner technologies is a major goal in green chemistry [1-2]. Among the several aspects of green chemistry, the catalytic amount of catalyst and the reduction or replacement of volatile organic solvents from the reaction medium is of at most importance [3- 5]. Polyethylene glycol (PEG) is more popular alternate reaction media, due to their properties like non-toxicity, bio-compatibility, and bio-degradability. Moreover, PEG is considered as a natural, inexpensive, safe, recyclable, degradable, non-flammable, facile, environmentally benign and abundantly available green solvent[6].C-C bonds formation reduction are important derivation in perfume, pharmaceutical, polymer applications [7-8]. These adducts are good Michael acceptors and can be used directly in the Diels-Alder reaction for further transformations also, the importance of Knoevenagel adducts has led to an exploration of several other reagent to promote knoevenagel condensation such as acid catalysts $CeCl_4.7H_2O/NaI$[8],$HClO_4$-SiO_2[9], $TiCl_4$[10] and $ZnCl_2$[11].Reports without any catalysts are also present with high temperature [12-14] ormicrowave irradiation [15] has been used for this transformations. Methylene active compounds carrying two electron withdrawing groups such as β-keto ester and acetylacetone are generally used in the known condensations. Very often, prolonged reaction condition or dangerous chemicals and high temperatures become a barrier for low field. Accordingly the development of a facile and efficient methodology for the knoevenagel condensation is desired. In this paper, we are describing our work in the successful use of potassium carbonateasa catalyst and PEG-400 as a green solvent at room temperatureforknoevenagelcondensationofaldehydeswithactivemethylenecompounds.

II. EXPERIMENTAL

The mixture of aldehyde (1mmol), Active methylene compounds (1mmol), and Potassium Carbonate (15mol%) in PEG as reaction media was taken in round –bottom flask at room temperature and stirred 60-70 min. After completion of the reaction (TLC), water was added stirred for a minute. The precipitate was filtrated and recrystallized from hot ethanol to obtain the pure product. Solvent used in TLC – Ethyl acetate and chloroform. Ratio – 1:3.

PRESENT WORK



R=Me, Ph, OEt
R'=Me, Ph, OEt, OMe
X= 4-NO2, 4-Me, 4-F, 4-OMe, 3-Cl, 2-F, Cinnamaldehyde, 2- OMe, 2-Cl, 4-Cl.

IJETIR

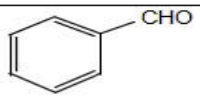
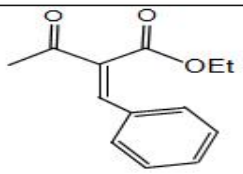
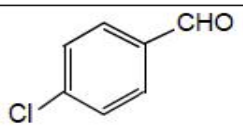
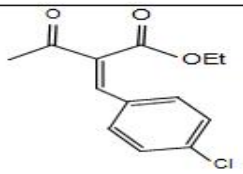
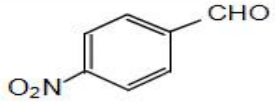
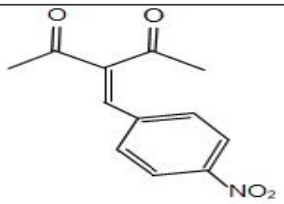
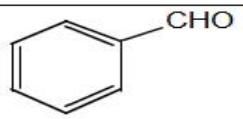
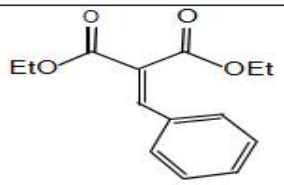
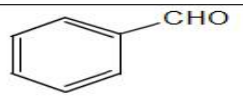
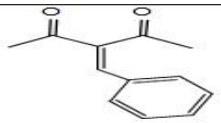
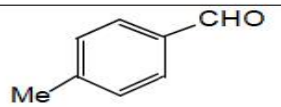
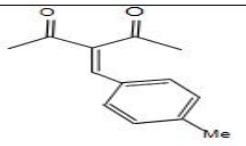
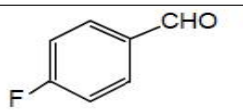
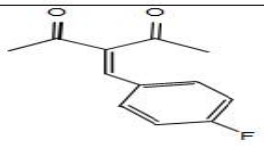
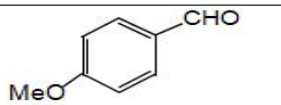
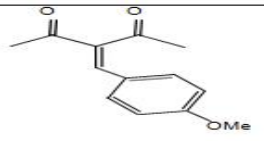
ISSN (Online) 2583-0554

International Journal of Emerging Technologies and Innovative Research (IJETIR)

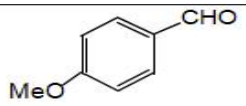
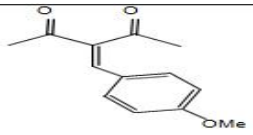
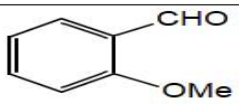
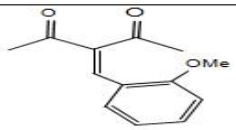
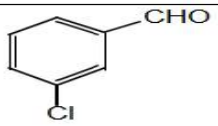
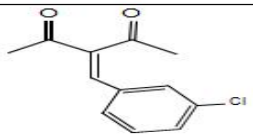
Volume 3, Issue 3, March 2023

Impact Factor: 5.731

Table: The Knoevengel condensation of β-keto ester or β-Diketones with aldehydes.

Entry	Aldehyde	Time (min)	Products	Yield (%)[a]	M.P. (°C)
1		70		92	60-62
2		60		91	87-89
3		60		93	169-170
4		60		93	52-54
5		70		93	54-56
6		60		83	80-82
7		60		88	90-92
8		60		81	80-84

IJETIR
ISSN (Online) 2583-0554
International Journal of Emerging Technologies and Innovative Research (IJETIR)
Volume 3, Issue 3, March 2023
Impact Factor: 5.731

8		60		81	80-84
9		60		87	69
10		60		86	85-86

The products were isolated and characterized by 1H NMR, 13C NMR and mass spectroscopy.

III. RESULT AND DISCUSSION

Apart of our program aimed at developing new selective and environment friendly methodologies, particularly C-C and C-N bond formation here we wish to present an efficient method for performing the knoevengel condensation between substituted aldehydes and β-diketones at room temperature and PEG-400 as a green solvent.The reaction is complete in 60-70 min. with a facile recovery of products in high yields (70- 93%).

ISOLATED YIELD (%)

Impressed with the result obtained, a few more examples were investigated for the generality of the reaction. Several aromatic and aliphatic aldehydes were treated with β-diketones to yield the corresponding condensation products. In this context aromatic aldehydes with electron withdrawing group afforded slightly higher yields compared to aromatic aldehydes bearing electron donating group.

IV. CONCLUSION

In conclusion, we have developed an economically and environment friendly catalysed for simple and efficient synthesis of trisubstituted alkenes in relatively short reaction times at room temperature condition. This involves the use of potassium carbonate as a very inexpensive and easily available catalyst under neutral and PEG-400 as a green solvent. Present methodology offers very attractive features such as reduced reaction times, higher yields and economics viability of the catalyst, when compared with other catalysts, which will have wide scope in organic synthesis.

REFERENCES

[1]. RajinderS.V,VasudevanV.N,PureAppl.Chem.2001;73:1309-1314.
[2]. Pasha, M.A, Jayashankara, V.P. Biorag. Med. Chem. 2007;17:621-623.
[3]. Pasha M. A. Manjula, K,jayashankara,V.P. Synth. React. Inorg. Met. Org Nano–Met. Chem.2006;36: 321-323;
[4]. Jones G; Org. React. 1967; 15:204-599.
[5]. Tietzc, L.E., Beifuss, U; In Comprehensive Organic Synthesis:Pergamon New York 1991; 2:314-394.
[6]. M. V. Reddy , J. S. Kim , K. T. Lim and Y. T. Jeong , Tetrahedron Lett., 2014, 55 , 6459 —6462
[7]. Bartoli ,G, Beleggia, R, Giuli S, Gialiani, A, Marcahtoni E,Massaccesi; M., Palett M; Tetrahedron Lett. 2006; 47:6501-6504.
[8]. Kantevari , S ; Bantu, R., Nagarapu, L; J. Mol. Catal.A :Chem. 2007;269:53-57.
[9]. LehnertW; Tetrahedron 1974; 30:301-305.
[10]. Shanthanrao, P.;Venkataratnam, R.V.; Tetrahedron Lett.1991;32:5821-5822.

[11]. Bigi, F. ;Conforti, M. L.;Maggi,R; Piiccino,A.;Sartori,G. ; Green Chem. 2000;2:101-103.

[12]. Hangarge, R.V.; Sonwange, S.A.; Jarikote, D.V.; Shingare, M .S. Green Chem. 2001: 3: 301-312.

[13]. Kaupp. G. ;Naimi – Jamal, M.R ., Schmeyers, J .; Tetrahedron 2003; 59:3753-3760.

[14]. Yadav , J. S.; Reddy , B.V. S.; Basak ,A.K.; Visali, B.; Narsaiah, A.V.; Nagaiah, K.Eur J. Org. Chem. 2004; 546-551.

[15]. Knoevenagel, L. Ber, 1898, 31,258.

[16]. (a) Siebenhaar, B, WO 9721659, 1977. (b) Hopp, R.; Thielmann, T.; Gottsch, W. US 521253, 1992. (c) Siebenhaar, B.; Casagrande, B. EP 0870750,1998.