International Journal of Trend in Scientific Research and Development (IJTSRD)
Volume 4 Issue 3, April 2020 Available Online: www.ijtsrd.com e-ISSN: 2456 – 6470

Sodium Hypophosphite: An Efficient Catalyst for the Synthesis of 2-Substituted Benzimidazole

Amit P. Tayade[1], Ramkrushna P. Pawar[2]

[1]Department of Chemistry, Dr. Babasaheb Ambedkar Marathwada University, Aurangabad, Maharashtra, India
[2]Department of Chemistry, Govt. Vidarbha Institute of Science and Humanities, Amravati, Maharashtra, India

ABSTRACT

The preparation of one-pot three components synthesis of 2-substituted Benzimidazole derivatives using reactants aldehyde o-phenylenediamines and sodium hypophosphite as catalysis in solvent medium. Reaction is carried out at 80^{0}C for 1 to 2 hour gives high yield.

KEYWORDS: *Benzimidazole, o-Phenylenediamine, sodium hypophosphite, Aldehyde*

How to cite this paper: Amit P. Tayade | Ramkrushna P. Pawar "Sodium Hypophosphite: An Efficient Catalyst for the Synthesis of 2-Substituted Benzimidazole" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-4 | Issue-3, April 2020, pp.641-643, URL: www.ijtsrd.com/papers/ijtsrd30622.pdf

IJTSRD30622

1. INTRODUCTION

Organic synthesis gives formation of heterocyclic compound. organic reactions in aqueous media have attracted much attention due its non toxic and unique reactivity and selectivity. Recently as per green chemistry concerned number of methods developed in organic synthesis, such as common heating method, reflux method. Solvent free synthesis, use of ionic liquids, microwave irradiation and ultrasound irradiation. All method are as concerned have their own merits and limitation.. Benzimidazole nucleus consist nitrogen in ring which shows numerous biological active compound and in several natural product such as antimicrobial (11), anti –hermitic (12), anti-HIV(13), antiulcer(14), etc. The simple and green protocol use for the synthesis of benzimidazole and its derivatives. One pot mono condensation of o- phenylenediamine and aldehyde in presence of sodium hypophosphite as catalyst gives the 2 substituted benzimidazole derivatives. Such heterocyclic compound that are wide interest because of their diverse biological and clinical application.

2. Experimental

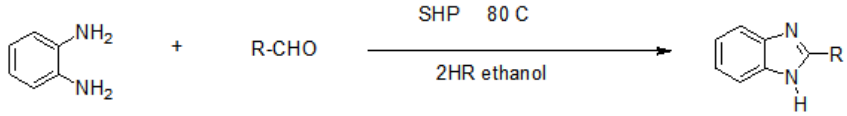
2.1. Experimental Section

All chemical were purchased from Merck, SD fine were commercially available and were used as received without further purification. Melting points were measured by open capillary method and were uncorrected. IR data collected on Agilent Cary 630 FTIR (range 4000-400). ^{1}HNMR Data recorded on Bruker Avance neo 500 NMR DMSO –d_6 MHz spectrometer

2.2. General method for synthesis of 2-benzimidazole:-

In one pot three component mono-condensation of o-phenylene diamine (3 mmol), aldehyde (3 mmol) sodium hypophosphite (SHP) (10 % mmol) as catalyst in solvent (15 ml) ethanol was stirred for about 2 hours at 80^{0}C, After the completion of the reaction (monitored by TLC). The reaction mixture was cooled to room temperature and poured on to crushed ice. The formed solid was filtered, washed with a mixture of ethyl acetate/hexane the crude products were purified by the crystallization from ethanol to afford the pure product **2-(-substituted)-1*H*-benzimidazole** All the products were confirmed by comparing their melting points, IR, ^{1}H NMR, ^{13}C NMR and LCMS-Mass spectral data with literature data.

Reaction:-



Scheme 1

Experimental table: 1 - One pot synthesis of 2-substituted Benzimadazole using catalyst SHP from various aldehydes

SR NO	Name of compound	melting point	% Yield
1	2-(-4-methoxylphenyl)-1H-benzimidazole	220-225	75
2	2(2-hydroxoyphenyl)-1H-benzimidazole	200-205	80
3	2(3–nitrophenyl)- 1H-benzimidazole	300-308	80
4	2-(4-chlorophenyl)- 1H-benzimidazole	290-295	80
5	2-(4-hydroxy-3methoxyphenyl)1Hbenzimidazole	230-240	70
6	2(4-di-methylaminophenyl)- 1H-benzimidazole	250-255	70
7	2-(4-methylphenyl)- 1H-benzimidazole	225-230	60

Table 2: Optimization of catalyst for synthesis of benzimidazole derivatives

Serial no	SHP,mol %	Percent yield	Time
1	No catalyst	10	120
2	5	60	120
3	10	80	120
4	20	70	120

Entry 04 :- Analytical data of 2-(-4-chlorophenyl)- 1H- benzimidazole
Yield - 80 %.
Melting point (^{0}C): 290-295^{0}C
Colour : Light yellowish powder

^{1}HNMR(500MHz, DMSO-d_6) :δ 12.85(s 1H N-H) , 8.20-8.13(2H dd), 7.66-7.51(2H dd), 7.20-7.19(1H s), 3.51, 2.52. ^{13}C NMR(125MHz, DMSO-d_6) :δ 150.28(-C=N), 143.76(C-N), 135.02, 134.75, 129.00,128.01, 122.57, 121.74, 118.85, 111.26,76.97, 56.35. FTIR (cm^{-1}):- 3447 (-NH), 3021(=CH), 1610(C=N), 1111(ether, C-O), ESI-MS: 225.79 m/z, Elemental analysis: $C_{14}H_{12}N_2O$; Calcd: C-74.98, H-05.39, N-12.49. ; Found: C-74.20, H-05.88, N-12.01

3. Result and discussion:-

In summary, a simple and efficient method has been developed for the synthesis of 2-substituted benzimidazole and its derivatives synthesis of by using sodium hypophosphite in ethanol as solvent through a mono-condensation in between aldehyde and o-phenylenediamines at 80^{0}C gives product. It was found that the catalyst gives good result in between 70% to 80% yield. Encouraged by these expected result, we have uses the same methodology on other aldehyde. The results as show in table -1

4. Conclusion:

In conclusion, this paper explain a convenient and efficient heating process for the synthesis of 2- substituted benzimidazoles by one pot reaction of o-phenylenediamine with aldehydes in presence of sodium hypophosphite in ethaonol. This method offers some advantages in terms of simplicity of performances in ethanol, low cast and follows along line of green chemistry. The catalyst is easy to handle, inexpensive and soluble in water.

5. Acknowledgement

The authors gratefully acknowledge the constant encouragement and support of the Head, Department of chemistry, Dr. BAMU Aurangabad. Principal, Deogiri College Aurangabad. We are also thankful SAIF Panjab University Chandigarh for analytical facilities.

6. Conflict of Interest:

The author have declared that no conflict of interest exists

References:

[1] Jeshma Kovvuri, Burri Nagaraju, Ahmed Kamal and Ajay K. Srivastava. An efficient of 2 –sub benzimidazole via plhotocatlystic condenstation of o-phenylenediamines and aldehyde.ACS Comb sci 2016.18,10,644-650

[2] Yeong Keng Yoon, Mohamed Ashrof Ali. Ang chee wei, Tan soo choon Rusli ismail, syntheisis and evaluation fo antimycobacterial activity ofnew benzimidazole aminoesters.european journal of med chemistry 2015,93 , 614-624

[3] Sathe B S,Jaychandran E. Jagtap V A, Deshmukh S D, synthesis and antifungal screening of fluoro benzothiazole imidazole derivatives Pharm.chem.2011, 3, 305-9.

[4] Heravi M M Bakhtiari. K. Oskooie H. A.Taheri S. J.Mol. Catal A.Chem,2007,263,279

[5] Manoj Takare, R. Shaikh , D Tayade, Catalyst free synthesis of 1,2- disubstituted benzimidazoles in aq mediaum using oxygen as the oxidant. Current chemistry letters 9,(2020) ,63-70.

[6] Naga Prashant K, K ravi Kumar. Green synthesis of benzimidazole derivatives: An overview of bulk drug synthesis.ijptr.2015, vol 8 no 9. Pp60-68

[7] Avinash Patil, swastika ganguly and sanjay surana, a systematic review of benzimidazole derivatives as an antiulcer agent. rasyan j. chem, 20081(3) :447-460

[8] Ashish kumar tewari and anil mishra, synthesis and antiviral activityes of N – substituted-2- substituted benzimidazole derivatives. Indian journal of chemistry vol 45B 2006.pp489-493

[9] Kabeer A. Shaikh and Vishal A. Patil An efficient solvent free synthesis of imidazolines and benzimidazoles using K4 [Fe(CN)6] catalysis. Org. Commun.2012, 5:1, 12-17

[10] Shatha Ibrahim Alaqeel . synthetic approaches to benzimidazoles from o-phenylenediamine: a literature review. Journal of Saudi chemical society.2017, 21, 229-237.

[11] A G Daimot, a c cremeux, JM hay, FP coulaud , Lancet,1983,17,652-656

[12] A. chimirri, S Grasso, AM Monforte, P Monforte, M Zappala. I farmaco, 1991, 46.925-933.

[13] K. Ishihara, T. Ichikawa, Y Komuro, S Ohara, K. Hotta, F. Arzneim. Drug. Res.1994, 44, 827-830.

[14] K. Kubo, YKohara , E. Imamiya Y. Sugiura .Y Inda, Furukawa, K Nishikawa, Naka, J med chem ,1993, 36, 2182-2195.

[15] Wiedemann, H Peli, Hjustus, S Adamus, V Brantl, H Lohmann, Arzneim forsch, Drug Res,1985, ,964-969