

Synthesis of β -Acetamido Ketones Catalysed by Red Mud from Kokan Region

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Abstract: Using readily available, non-corrosive, and environmentally friendly catalysts like Kokan Red Mud, the multicomponent condensation of arylaldehyde-acetyl chloride, acetonitrile, and an enolizable ketone was studied as a one-pot synthesis of β -acetamidoketones with high yields. Kokan Red Mud was the catalyst with the highest level of effectiveness. The technique was used in straightforward experimental conditions, and the relatively short reaction times and high yields show that it is a very useful method for the production of β -acetamidoketones on a wide scale.

Keywords: Red Mud from the Kokan Region - Dawson; Dakin-West; β -Acetamido Ketone; Heterogeneous Catalysis.

I. INTRODUCTION

For the synthesis of incredibly complex chemical compounds, multicomponent reactions (MCRs) have proven to be a highly effective and potent method. [1] They are additionally utilised to produce organic chemical libraries that are used in drug development. Over the past ten years, a wide range of synthetic routes in organic chemistry have been investigated using multicomponent synthesis methods [3-5]. MCRs are effective and inexpensive reactions that have been used for parallel and combinatorial synthesis using templates that are significant from a pharmacological standpoint [6,10]. Multicomponent reactions in organic synthesis [11, 12] allow the simultaneous formation of two or more bonds and the combining of more than two building blocks in a practical, time-saving one-pot process.

The Dakin-West reaction is a very efficient way to create different acetamidoketones. He created a second novel technique for the synthesis of β -acetamidoketones by MCR of acetophenone, arylaldehyde, and acetyl chloride in acetonitrile using different catalysts. CoCl_2 and other catalysts and reagents have been thoroughly studied for these reactions. Montmorillonite K-10 clay, FeCl_3 , $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$, $\text{Sc}(\text{OTf})_3$, $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, $\text{Fe}(\text{HSO}_4)_3$, sulfuric acid supported on silica, H3PW12O40, and Amberlyst-[13-15].

II. MATERIALS AND MEYHODS

All research substances were purchased from S.D. FINE. Using the melting points of the Thiles tube and paraffin, the melting points of the produced compounds were computed. All ^1H NMR spectra, unless otherwise stated, were obtained in CDCl_3 using a Bruker Advance DPX 200/300 MHz spectrometer using TMS as the internal standard. To separate the chemicals in the system, stationary phases made of 60 and 120 mesh silica gel were utilised. [16-18]

III. EXPERIMENTAL TECHNIQUE

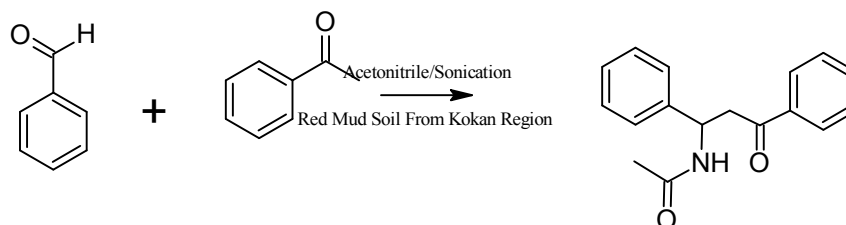
We have created a brand-new, ecologically friendly process for producing β -acetamidocarbonyl compounds using Kokan Red Mud as a reagent.

General steps for producing β -Acetamido Ketones using a Green Approach

10 mmol of ketone and 10 mmol of aldehyde were diluted in 10 mL of acetonitrile. The reaction mixture received 15 mg of Kokan Region Red Mud as a catalyst. The process was sonicated at 40 degrees Celsius for three to six hours, depending on the reactants. TLC was used to examine the reaction's progression. The crude reaction mixture was then produced by rotary evaporating acetonitrile after the reaction had finished. This unfinished mixture was treated with 10

ml of ethyl acetate and washed 2-3 times with water before being collected in a beaker. The organic layer (ethyl acetate layer) was evaporated under decreasing pressure after drying on sodium sulphate. The chemicals were purified using flash column chromatography and silica gel.

Reaction Scheme:



Observation Table 1 for synthesis of -Acetamido Ketones

Sr. No.	Aldehyde	Ketone	% Yield	Physical Constant 0 C	Reaction Time in hrs
1	benzaldehyde	1-phenylethanone	88	103	3.2
2	3-chlorobenzaldehyde	1-phenylethanone	95	105	2.9
3	4-chlorobenzaldehyde	1-phenylethanone	90	146	3.0
4	4-nitrobenzaldehyde	1-phenylethanone	95	152	3.0
5	2-chlorobenzaldehyde	1-phenylethanone	92	133	3.0
6	4-bromobenzaldehyde	1-phenylethanone	94	144	3.0
7	3-bromobenzaldehyde	1-phenylethanone	96	102	3.1
8	3-nitrobenzaldehyde	1-phenylethanone	95	140	3.1
9	2-nitrobenzaldehyde	1-phenylethanone	93	188	3.0

IV. RESULTS AND DISCUSSION

Step-by-step, conventional procedures were used to accomplish the reaction. Red Mud from the Kokan region was used as a catalyst for the environmentally friendly synthesis of several -Acetamido ketones derivatives. Utilizing ketone (1 equiv.) and aldehyde (1 equiv.), Red Mud from the Kokan Area in acetonitrile solvent, and heating in sonication at 400C for 3-6 hours, the necessary chemicals were created. Using different substituted aldehydes, ketones, and acetonitrile under ideal reaction conditions, many -acetamido ketones molecules were synthesised. The obtained yields ranged between 85 and 96%. This approach generates a better yield and purity with no byproducts; it is environmentally friendly.

V. CONCLUSION

This paper describes a straightforward and effective method for the synthesis of -acetamidoketones via the four-component coupling of aromatic aldehydes, an enolizable ketone, and acetyl chloride in acetonitrile using Red Mud from Kokan Region as a low-cost, commercial, non-corrosive, and environmentally benign catalyst with an ecological approach.

The presented approach has a wide range of applications in organic synthesis due to its advantageous characteristics, which include a straightforward procedure coupled with relatively simple workup, short reaction durations, and

excellent yields. This approach is strongly suggested as a practical and alluring method for the large-scale synthesis of -acetamidoketones.

REFERENCES

- [1]. (a) Tietze, L. F.; Brasche, G.; Gericke, K. M.; Wiley-VCH Verlag GmbH & Co. KGaA: Weinheim, 2006.
- [2]. (a) Barry, B. T.; Dennis, G. H., *Chem. Rev.* 109, 4439, (2009).
- [3]. Strubing D, Neumann H, Klaus S, Hubner S, Beller M., *Tetrahedron*, 61: 113-33, (2005).
- [4]. Heydari A, Arefi A, Khaksar S, Shiroodi R K., *J Mol Catal A*, 271: 142, (2007)
- [5]. Guillena G, Ramon D J, Yus M., *Tetrahedron: Asymmetry*, 18: 693, (2007)
- [6]. Hulme C, Peng J, Tang S-Y, Burns C J, Morize I, Labaudiniere R., *J Org Chem*, 63: 8021, (1998)
- [7]. Hulme C, Ma L, Romano J, Morrissette M., *Tetrahedron Lett*, 40: 7925, (1999)
- [8]. Zhu J, Bienayme H. *Multicomponent Reactions*. Weinheim, Wiley, 2005
- [9]. Nitin A. Mirgane, Vitthal S. Shivankar, Sandip B. Kotwal, Gurumeet C. Wadhawa, Maryappa C. Sonawale, Degradation of dyes using biologically synthesized zinc oxide nanoparticles, *Materials Today: Proceedings*, Volume 37, Part 2021, 849-853, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.06.037>.
- [10]. Nitin A. Mirgane, Vitthal S. Shivankar, Sandip B. Kotwal, Gurumeet C. Wadhawa, Maryappa C. Sonawale, the Waste pericarp of ananas comosus in green synthesis zinc oxide nanoparticles and their application in wastewater treatment, *Materials Today: Proceedings*, Volume 37, Part 2, 2021, 886-889, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.06.045>.
- [11]. Shubhada S. Nayak, Nitin A. Mirgane, Vitthal S. Shivankar, Kisan B. Pathade, Gurumeet C. Wadhawa, Adsorption of methylene blue dye over activated charcoal from the fruit peel of plant hydnocarpuspentandra, *Materials Today: Proceedings*, Volume 37, Part 2, 2021, 2302-2305, ISSN 2214-7853, <https://doi.org/10.1016/j.matpr.2020.07.728>.
- [12]. Patil, D.D.; Mhaske, K.D.; Wadhawa, C.G., Antibacterial and Antioxidant study of *Ocimum basilicum* Labiatae (sweet basil), *Journal of Advanced Pharmacy Education & Research* (2011) 2, 104-112.
- [13]. Dinanath PD, Gurumeet WC, 2013. Antibacterial, antioxidant and anti-inflammatory studies of leaves and roots of *Solanum xanthocarpum*. *Unique J Ayurvedic Herb Med* (2013) ;(3):59-63.
- [14]. Patil DD, Mhaske DK, Wadhawa GC. Green Synthesis of 3,4-dihydropyrimidinone using ferrous sulphate as recyclable catalyst, *Journal of Pharmaceutical Research and Opinion*, 2011; 1(6): 172-174.
- [15]. Nayak, Shubhada S., et al. "Green synthesis of the plant assisted nanoparticles from *Euphorbia neriifolia* L. and its application in the degradation of dyes from industrial waste." *Plant Science Today* 8.2 (2021): 380-385.
- [16]. Algul, O.; Karabulut, A.; Canacankatan, N.; Gorur, A.; Sucu, N.; Vezir, O. Apoptotic and anti-angiogenic effects of benzimidazole compounds: Relationship with oxidative stress mediated ischemia/reperfusion injury in rat hind limb. *Antiinflamm. Antiallergy Agents Med. Chem.* 2012, 11, 267-275. [CrossRef]
- [17]. Thakuria, H.; Das, G. An expeditious one-pot solvent-free synthesis of benzimidazole derivatives. *Arkivoc* 2008, 15, 321-328. [CrossRef]
- [18]. Rithe, S.R.; Jagtap, R.S.; Ubarhande, S.S. One Pot Synthesis of Substituted Benzimidazole Derivatives and Their Characterization. *RASAYAN J. Chem.* 2015, 8, 213-217.