IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429
Volume 6, Issue 5, June 2026
Impact Factor: 8.2

Red Mud Catalyzed Microwave Assisted Beckman Rearrangement Reaction

Rohit Patil[1], Pranali Satpute[2], Abhishek Yadav[3], Sajid F. Shekh[4], Yashwant C. Gaikawad[5], Maryappa C Sonawale[6]

[1,2]Student P.G.Department of Chemistry Veer Wajekar College Phunde , Uran
[3]Student U.G.Department of Chemistry Veer Wajekar College Phunde , Uran
[4]Incharge Principle of Anjuman Islam College, Murud
[5,6]Assistant Professor Department of Chemistry Veer Wajekar College Phunde , Uran

Abstract: *One of the best method for the prepartion of the amide is the beckman rearragment reaction.In this reaction oximes are converted to amide.this is Acid-catalyzed oxime to amide reaction called the Beckmann rearrangement is a rearrangement. German scientist Ernst Otto Beckmann (1853-1923) .Various acid catalysts are used to synthesize the amides from oxime.Amide Derivatives are very widely in the industry. In this research we prepared the different amides using aromatic aldehydes under green and mild conditions and without additional organic solvents using the redmud as catalyst . Beckmann rearrangement of ketoximes was performeddifferently using mortar pestle andsoil (Red mud) consistingof various salt. Especially for the conversion of Keto oxime to Amide.*

Keywords: Red mud Catalyzed Microwave Assisted Beckman Rearrangement Reaction

I. INTRODUCTION

The Beckmann rearrangement is frequently utilized in organic chemistry and its branches; for example, the Preparation of Ɛ-caprolactam from cyclohexanone oxime is employed in great-scale manufacturing of polymers Nylon-6. The Beckmann arrangingcould be aspecificrearrangement of anisotropic type and studied by different Lewis and Bronsted acids.It has commercialsignificancewithin theadaptation of ketone and cyclohexanone organic compounds form Ɛ-caprolactam and catalysts for this particular application have usually been described.Though, a fewstudies of the mechanism and dynamicsof the gasfraction reaction have appeared in writing. Used fora few decades, we've used atomic number 13 and orthophosphates [3]and differentconnected systems as Lewis acid catalysts in severalion organic processes with reagents in the vapor stage [4-6]. The Beckmann rearrangement is in the middle of these [7-9]. Mostly this reaction is governed by the acid catalyst. severalattemptsto exchangevitriol with a solid acid catalyst aremade in this reaction many times, the Beckmann rearrangement is meted out in powerfully acidic and dehydrating media reminiscent of phosphorus pentachloride, focusedchemical element acid, or the alleged 'Beckmann's mixture' that contains carboxylic acid, anhydride, and range chemical. [10] Since different reagents aren'tappropriate for an outsized number of sensitive substrates, many attempts have been made to realize the Beckmann wasarrangingbelowconsiderablysimpler conditions. In these studies, SOCl2, [11] silicon oxideas gel, [12] metallic elementoxide on supported on solid silica gel, [13] soil like montmorillonite and KSF, [14]$BiCl_3$, [15] 2,4,6-trichloro-1,3,5 triazine, [16] GaCl3[17] Phosphorus Pent oxide , [18] Chloro sulphonic acid , [19] Phosphorus Pent chloride , [20] aluminiferous as Lewis acid [Rhodium Cl(cod)]2, [21] Yetterbitium(OTf)3, [22] RuthiumCl3, [23] Mercury Chloride , [24] many liquid-phase contact actionsystem, reminiscent ofacid sulphamic, [25] diffeentchlorosulphonic acid, [26] pure anhydrous oxalic acid, [27] ethyl groupand formatesetherate of trifluoride, [28] bis 2-oxo- 3-oxazolidinyl in element chloride [29] and diethyl chlorophosphate [30] potential solid acid catalysts such as zeolite with MFI , [31] medium-pore silicoaluminophosphate molecular sieve with tunable acidity, [32] mesoporous silica, [33] Ytterbium , [34] Zeolite Socony Mobil–5, [35] SiO2/Al2O3 Molar Ratio of 50, [36] TiO2-ZrO2 dioxide , [37] Associate in Nursingd mordenite, [38] areevaluate as substitute for the historicallyused reagents. Recently, manyWith

DOI: 10.48175/IJARSCT-35621

IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429 Volume 6, Issue 5, June 2026 Impact Factor: 8.2

the catalyst from the [with La(III), Sm(III) and Ce(III), in the middle of others] have conjointly been found to result in the arranging. [39, 40] The reaction may be performed within the vapor stage, which is applicable even on an industrial scale with the employment of a high-silica f MFI zeolite.mineral as the catalyst. [41] it'stoopossible to initiate the Beckmann rearrangement in seriousaqueous [42] ionic liquids as a catalyst. [43]

Red mud, produced using the Bayer process, is a business waste acquiredat some point in the manufacturing of Al. For each ton of alumina saltformed, about 1.65lots of crimson as mud is launched.it's milespredictedwithgreater than 66 millionlots of wasteyearlyworldwide [44,45]. The crimsondust is commonlysent in sea water[46] or disposed into a land filing [47] pollutingthe encirclingwater, along with soil and atmosphere, particularlywithinside theregionsin which this enterprise is situated [48]. So, steps ought to be used to recycle the particular waste in a green way or better. Basedon theRed mud composition, it may be reused and utilized inseveralscientific fields. For example, it may be used as a recovery of metals or may be used as a capacityopportunity catalyst because it, in particular, includes an aggregate of oxides of Fe, Al, and Ti and a smaller quantity of Silicon, Calcium, andsodium [49-50]. Red mudalso can act as a pigment because its iron-wealthylevels have a crimson color [51]. However, a largequantity of crimsondustwishes to be recycled and greaterpowerfulprogramsmust be developed. In this method,We have used Red dust as the catalyst for the Beckman Reaction; this responsebecame Carried the use oftraditionaltechniquesin conjunction withinexperiencedtechniques.

II. EXPERIMENTAL

Reactions were carried out
in a nitrogen environment, and TLC silica gel plates (60 F254) wereusedtomonitor them. All solvents and reagents wer e purchased from S.D.Fine chemicals. The oximeswere synthesized using normal techniques, and their purity was deter mined by melting point before use. Standard 1H and 13C NMR measurements were conducted on asolvent like $CDCl_3$ using Trimethylsilaneas an internal standard. The IR spectra were captured using a Scimandzu IR 2000 class FT IR spectrometer. Shimadzu GCMS Q 5050A low-resolution mass spectra were acquired; the Frgmentation pattern is presented following the matching m/z valueof compounds.

General procedure:

Microwave Approach

In a glass vessel, a combination of ketoxime (10 mmoles) and anhydrous red mud (20 mg) was cooked in a microwave at 160 °C. The reaction started with effervescence and took 3-6 minutes to finish (TLC check). 10 ml of water and 10 ml of diethyl ether were added to the Dark brown molten mass after reaching 20^0C . Solid $NaHCO_3$ was added to the reaction mixture to neutralize it. Use 10 x 2 ml of Diethyl ether to remove the aqueous layer from the organic layer. Ether extracts were combined and water was removed using a dehydrating agent like sodium sulphate before being isolated in a vacuum. The product was refined using a chloroform and methanol solvent solution on a 300 meshsilica gel to obtain the matching amide in good yield and purity.

Observation

Table 1:

Final Product	Melting Point	Reaction Time (Microwave)	% Yield
Acetanilide	114-115 0c	6 min	91
Para nitro Acetanilide	215-217 0c	6 min	94
Para bromo acetanilide	165-168 0c	6 min	96
Para chloroacetanilide	171-178 0c	6 min	95
Benzmide	127-130 0c	5 min	95
Para nitro Benzmide	199-201 0c	6 min	88

IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429 Volume 6, Issue 5, June 2026 Impact Factor: 8.2

Para bromo Benzmide	190-193 0c	6 min	90
Acetamide	79-82 0c	6 min	91
Benzanilide	160–162 °C	6 min	88
N-(4-Methylphenyl)acetamide	148–149 °C	6 min	90
Propionanilide	108-109°C	5 min	91
Butyranilide	98–99 °C	6 min	94
N-(2-Thiophenyl)acetamide	152–154°C		

Observation Table 2

Conc. of Catalyst

Conc. of Catalyst (For Acetanilide Synthesis)	**Reaction Time (Microwave)**	**Reaction % Yield (Microwave)**
1 mg	15 min	60
3 mg	12 min	70
5 mg	10 min	80
7 mg	8 min	80
10 mg	6 min	95
12 mg	08 min	90
15 mg	08 min	90

Observation Table 3

Recycling of Catalyst

Cycle of the Catalyst	**Reaction Time (Microwave)**	**Reaction % Yield (Microwave)**
First Cycle	6 min	94
Second Cycle	6 min	93
Third Cycle	6 min	90
Fourth Cycle	7 min	88
Fifth Cycle	7 min	88

Spectral analysis

1) Acetanilide : M.p. 114–115 °C

IR in Cm ¯1 :3290Cm ¯1, 2850Cm ¯1, 1660Cm ¯1, 1590 Cm ¯1, 1510 Cm ¯1, 919 Cm ¯1, 858 Cm ¯1, 750 cm−1;
1H NMR(300 MHz, CDCl3): δ 2.12 (3H, s), 7.15 (1H, t,), 7.34 (2H,m), 7.53 (2H, m), 8.37 (1H, s);

2) Benzanilide : M.p. 160–162 °C

IR: cm ¯1: 3340cm ¯1, 3050cm ¯1, 1650cm ¯1, 1600 cm ¯1, 1539 cm ¯1, 1520 cm ¯1, 1440cm ¯1, 1320cm ¯1, 928cm ¯1, 797cm ¯1, 725 cm ¯1, 717cm−1;
1H NMR (300 MHz, CDCl3): δ 7.10–7.27 (1H, m), 7.32–7.50(2H, m), 7.51–7.78 (3H, m), 7.62–7.68 (2H, m), 7.70–7.82 (2H, m),7.87 (1H, s);

3) N-(4-Methylphenyl)acetamide: M.p. 148–149 °C

IR cm-1: 3290cm-1, 3120 cm-1, 2824cm-1, 1686cm-1, 1624cm-1, 1484cm-1, 828cm-1,758 cm-1;
1H NMR (300 MHz, CDCl3): δ 2.10 (3H, s), 2.38 (3H, s),7.16 (2H,), 7.37 (2H,), 8.31 (1H, s)

IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429 Volume 6, Issue 5, June 2026 Impact Factor: 8.2

4) Propionanilide:M.p. 108–109 °C
IR cm-1: 3232cm-1, 2908 cm ¯1, 2944cm ¯1, 1688cm ¯1, 1612cm ¯1, 1566cm ¯1, 1486cm ¯1, 1450cm ¯1, 917cm ¯1, 761cm ¯1,681 cm−1.
1H NMR (300 MHz, CDCl3): δ 1.24 (3H, t,), 2.37(2H, q,), 7.17 (1H, t), 7.18 (2H, t,),7.72 (2H, d,), 7.88 (1H, s)

5) Butyranilide :M.p. 96–98 °C
IR cm-1:3264cm-1, 2971cm-1, 2760cm-1, 1669cm-1, 1509cm-1, 1510cm-1, 900cm-1, 760cm-1, 691 cm^{-1};
1H NMR(300 MHz, CDCl3): δ 0.90 (3H, t,), 1.68 (2H, m), 2.20 (2H,), 7.18 (1H, m), 7.17 (2H, t,), 7.34 (2H, d), 7.80 (1H, s);

6) N-(2-Thiophenyl)acetamide M.p. 152–154 °C.
IR cm-1: 3164cm-1, 2871cm-1, 2750cm-1, 1679cm-1, 1519cm-1, 1520cm-1, 990cm-1, 770cm-1, 681 cm^{-1};
1H NMR(300 MHz, CDCl3): δ 2.14 (3H, s), 6.83 (1H, m), 6.79 (2H,m), 10.59 (1H, s);

III. RESULTS AND DISCUSSION

To make acetanilide, acetophenone oxime (10 mmoles) (Table 1, Entry 1) and Red mud (10 mg.) were microwaved for six minutes (93 percent yield). We investigated the influence of the quantity of red mud on the Beckmann rearrangement of the different keto oxime to determine the best reaction conditions. It was discovered that 10 mg of red mud was enough to provide identical outcomes. However, using less than 10 mg of red mud led to a low product yield and a low recovery of the starting material. This result revealed that 10 mg of red mud was sufficient to convert the starting material completely. Various ketoximes were treated to the reaction to test its generality and scope.

IV. CONCLUSION

Finally, we have demonstrated that Red mud was employed as an acid catalyst that is both environmentally acceptable and effective. This catalyst is a more practical alternative to the described catalysts due to its operational simplicity, use of commercially accessible waste used as the catalyst, solvent-free reaction state, quick reaction time, fast set-up, and good yield. In a short time, it effectively catalyzes oxime to amide as beckman rearrangement of a variety of ketoximes into matching amides in a solvent-free environment in miceowaveapproch. More research is being done to broaden the to form the amide in different organic syntheses.

REFERENCES

[1] J. D. BUTTERan d T. C. POLESJ. . Chem. Soc. Perkin II,41 (1973).
[2] K. L. SAMARINR.. I. POMILENKaOnd, I. YA. TYURGAEV.Kinet. Katal. 16, 8% (1975).
[3] K. K. KEARBYA. ctes Con-a . Int. Catal. 2OParis, 2, 2267(1960).
[4] J. M. MARlN~sand**A .**ALBEROLAA.n . Quim. 67,37(1971).
[5]J. M. MARINASA. . ALBEROLAan,d M. S. ESTEBANA. n.Quim. 68, 1295 (1972).
[6] A. ALBEROLAM. . S. ESTEBANJ.. M. MARINASan, d R. PEREZ-OSSORIAOn. . Quim. 70,944 (1977).
[7]A. ALBEROLAM, . BLANCO,M . S. ESTEBANJ. .M.MARINASa,n d R. PEREZ-OSSORIOA.n . Quim. 72, 887(1976).
[8] M. S. ESTEBANan d J . M. MARINASA. n. Quim. 74, 1413(1978).
[9] A. COSTA. M. S. ESTEBANJ. . M. MARINASa,n d R.PEREZ-OSSORIAOn.. Quim. 73, 1521 (1977).
[10] JB Hendrickson; DJ Cram; GS Hammond, *Organic Chemistry*, 3rd ed., McGraw-Hill: Tokyo, **1970**, 708.
[11]MB Smith; J March, *Advanced Organic Chemistry*, 6th ed.; John Wiley & Sons: Hoboken, **2007**, 1613.
[12] RN Butler; ODA Donoghue, *J. Chem. Res., Synop.,* **1983**, 18.
[13] A Costa; B Mestres; JM Riego, *Synth. Commun.,* **1982**, 12, 1003.
[14]MK Dongare; VV Bhagwat; CV Ramana; MK Gurjar, *Tetrahedron Lett.,* **2004**, 45, 4759.

[15] HM Meshram, *Synth. Commun.,* **1990**, 20, 3253.
[16]AJ Thakur; A Boruah; D Prajapati; JS Sandhu, *Synth. Commun.,* **2000**, 30, 2105.
[17]LD Luca; G Giacomelli, AJ Porcheddu, *J. Org. Chem.,* **2002**, 67, 6272.
[18]P Yan; P Batamack; GKS Prakash; GA Olah, *Catal. Lett.,* **2005**, 103, 165.
[19]H Sato; H Yoshika; Y Izumi, *J. Mol. Catal., A: Chem.* **1999,** 149, 25.
[20]D Li; F Shi; S Guo; Y Deng, *Tetrahedron Lett.,* **2005,** 46, 671.
[21] J Peng; Y Deng, *Tetrahedron Lett.,* **2001,** 42, 403.
[22]M Arisawa; M Yamaguchi, *Org. Lett.* **2001,** 3, 311.
[23] JS Yadav; BVS Reddy; AV Madhavi; YSS Ganesha, *J. Chem. Res.*, Synop. **2002,** 236.
[24]SK De, *Synth. Commun.,* **2004,** 34, 3431.
[25]C Ramalingan; Y T Park, *J. Org. Chem.,* **2007,** 72, 4536.
[26]LD Luca; G Giacomelli; A Porcheddu, *J. Org. Chem.*, **2002**, 67, 6272.
[27]Y Furuya; K Ishihara; H Yamamoto, *J. Am. Chem. Soc*., **2005**, 127, 11240.
[28]S Chandrasekhar; K Gopalaiah, *Tetrahedron Lett*., **2002**, 43, 2455.
[29]S Antikumar; S Chandrasekhar, *Tetrahedron Lett*., **2000**, 41, 5427.
[30]M Zhu; C Cha; W Deng; X Shi, *Tetrahedron Lett*., **2006**, 47, 4861.
[31]AR Sardarian; Z Shahsavari-Fard; HR Shahsavari; Z Ebrahimi, *Tetrahedron Lett.,* **2007**, 48, 2639.
[32]A Thangaraj; S Sivasankar; P Ratnasamy, *J. Catal.,* **1992,** 137, 252.
[33]PS Singh; R Bandyopadhyay; SG Hegde; BS Rao, *Appl. Catal. A,* **1996,** 136 249.
[34]D Shouro; Y Moriya; T Nakajima; S Mishima*, Appl. Catal. A,* **2000,** 198, 275.
[35]A Corma; H Garcia; J Primo, *Zeolites,* **1991**, 11 593.
[36]T Takahashi; MNA Nasution; T Kai, *Appl. Catal. A,* **2001,** 210, 339.
[37] JS Reddy; R Ravishankar; S Sivanker; P Ratnasamy, *Catal. Lett.,* **1993,** 17 139.
[38]DS Mao; GZ Lu; QL Chen; ZK Xie; YX Zhang, *Catal. Lett.,* **2001,** 77, 119.
[39]PS Landis; PB Venuto, *J. Catal.,* **1996,** 6, 245.
[40]B Thomas; S Sugunan, *Microporous Mesoporous Mater.,* **2006**, 96, 55.
[41]SV Priya; JH Mabel; M Palanichamy; V Murugesan, *Stud. Surf. Sci. Catal.,* **2008**, 174, 1147.
[42]Y Izumi; H Ichihashi; Y Shimazu; M Kitamura; H Sato, *Bull. Chem. Soc. Jpn.,* **2007**, 80, 1280.
[43]M Boero; T Ikeshoji; CC Liew; K Terakura; M Parrinello, *J. Am. Chem. Soc.,* **2004**, 126, 6280
[44]S. Patel and B. K. Pal, "Current status of an industrial waste: red mud an overview," *Ijltemas*, vol. 4, no. 8, pp. 1–6, 2015.View at: Google Scholar
[45]J. Ortega, M. Cabeza, A. Tenza-Abril, T. Real-Herraiz, M. Climent, and I. Sánchez, "Effects of red mud addition in the microstructure, durability and mechanical performance of cement mortars," *Applied Sciences*, vol. 9, no. 5, p. 984, 2019.View at: Publisher Site | Google Scholar
[46] V. Dethlefsen and H. Rosenthal, "Problems with dumping of red mud in shallow waters. A critical review of selected literature," *Aquaculture*, vol. 2, pp. 267–280, 1973.View at: Publisher Site | Google Scholar
[47] H. Zhang, H. Li, Y. Zhang, D. Wang, J. Harvey, and H. Wang, "Performance enhancement of porous asphalt pavement using red mud as alternative filler," *Construction and Building Materials*, vol. 160, pp. 707–713, 2018.
[48] S. Baseden and D. Grey, "Environmental study of the disposal of red mud waste," *Marine Pollution Bulletin*, vol. 7, no. 1, pp. 4–7, 1976.View at: Publisher Site | Google Scholar
[49]Y. Çengeloğlu, E. Kir, and M. Ersöz, "Recovery and concentration of Al(III), Fe(III), Ti(IV), and Na(I) from red mud," *Journal of Colloid and Interface Science*, vol. 244, no. 2, pp. 342–346, 2001.
[50] S. Sushil and V. S. Batra, "Catalytic applications of red mud, an aluminium industry waste: a review," *Applied Catalysis B: Environmental*, vol. 81, no. 1-2, pp. 64–77, 2008.View at: Publisher Site | Google Scholar
[51] J. Carneiro, D. M. Tobaldi, W. Hajjaji et al., "Red mud as a substitute coloring agent for the hematite pigment," *Ceramics International*, vol. 44, no. 4, pp. 4211–4219, 2018.