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Jumlah Penulis	:	Oktavianty)	ggoro	, Luqman Buchori, Setia Budi Sasongko, Herawati			
Status Pengusul	:	penulis ke-3					
Identitas Jurnal Ilmiah	:	a. Nama Jurnal	1	Bulletin of Chemical Reaction Engineering & Catalysis			
		b. Nomor ISSN	:	1978-2993			
		c. Vol, No., Bln Thn	1	Volume 14 Issue 3 Year 2019			
		d. Penerbit		Diponegoro University			
		e. DOI artikel (jika ada)		https://doi.org/10.9767/bcrec.14.3.4248.678-682			
		f. Alamat web jurnal					
		5		https://ejournal2.undip.ac.id/index.php/bcrec/article/view/4248/ https://ejournal2.undip.ac.id/index.php/bcrec/article/view/4248/			
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Reviewer 2

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Prof. Ir. Abdullah, M.S., Ph.D. NIP. 195512311983031014 Unit Kerja : Dept. Teknik Kimia FT UNDIP

Semarang, Juni 2020

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Reviewer 1

Prof. Dr. Ir. Bakti Jos, DEA NIP. 196005011986031003 Unit Kerja : Dept. Teknik Kimia FT UNDIP

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Status Pengusul : penulis ke-3 Identitas Jurnal Ilmiah : a. Nama Jurnal : Bulletin of Chemical Reaction Engineering & Catalysis b. Nomor ISSN : 1978-2993 : 1978-2993 c. Vol, No., Bln Thn : Volume 14 Issue 3 Year 2019 d. Penerbit : Diponegoro University e. DOI artikel (jika ada) : https://doi.org/10.9767/bcrec.14.3.4248.678-682 f. Alamat web jurnal : https://ejournal2.undip.ac.id/index.php/bcrec/article/view/4248 Alamat Artikel : https://ejournal2.undip.ac.id/index.php/bcrec/article/view/4248/ 2671 g. Terindex : Scopus, Q3 Kategori Publikasi Jurnal Ilmiah (beri ✓ pada kategori yang tepat) : Jurnal Ilmiah Internasional Jurnal Ilmiah Nasional Terakreditasi Jurnal Ilmiah Nasional Tidak Terakreditasi 								
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Reviewer 2

Prof. Ir. Abdullah, M.S., Ph.D. NIP. 195512311983031014 Unit Kerja : Dept. Teknik Kimia FT UNDIP



e-ISSN: 1978-2993 Bulletin of Chemical Reaction Engineering & Catalysis

Volume 14, Issue 3, Year 2019 (Regular Issue: December 2019)

Available online since 30th September 2019 at: https://bcrec.undip.ac.id/ or https://bcrec.id

Bull. Chem. React.	No. 3	Pages:	Semarang,	e-ISSN:
Eng. Catal. Vol. 14		478 - 714	December 2019	1978-2993



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Research Article

Basicity Optimization of KF/Ca-MgO Catalyst using Impregnation Method

Didi Dwi Anggoro*, Luqman Buchori, Setia Budi Sasongko, Herawati Oktavianty

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> Received: 25th January 2019; Revised: 11th May 2019; Accepted: 20th May 2019; Available online: 30th September 2019; Published regularly: December 2019

Abstract

This research aimed at determining the optimum value between calcination temperature (X_1) , calcination time (X_2) and %wt KF (X_3) toward optimum basicity of KF/Ca-MgO catalyst. Approximately 2-4%wt KF was added to the KF/Ca-MgO catalyst using the impregnation method to assist the Ca-MgO, at 450-550 °C and a calcination time of 2-4 hours. Furthermore, its basicity was analyzed using Tanabe's titration method. The use of Variance Analysis (ANOVA), indicated that calcination temperature (X_1) factor achieved the highest basicity of KF/Ca-MgO catalyst, as indicated by its high *F*-value (16.46262) and low *p*-value (0.0067). The correlation between each operating variables and the responses were shown in a mathematical equation. The optimization value is estimated by limiting the calcination temperature from 415.9 to 584.1 °C, with a calcination time ranging from 1.32 to 4.68 hours, and %wt KF of 1.3182 to 4.6818 % that obtained 1.18 mmol/g for the optimal catalyst basicity. Copyright © 2019 BCREC Group. All rights reserved

Keywords: KF/Ca-MgO catalyst; Basicity; Optimization; Response Surface Methodology

How to Cite: Anggoro, D.D., Buchori, L., Sasongko, S.B., Oktavianty, H. (2019). Basicity Optimization of KF/Ca-MgO Catalyst using Impregnation Method. *Bulletin of Chemical Reaction Engineering & Catalysis*, 14(3): 678-682 (doi:10.9767/bcrec.14.3.4248.678-682)

Permalink/DOI: https://doi.org/10.9767/bcrec.14.3.4248.678-682

1. Introduction

Potassium Fluoride (KF) is an alkaline halide molecule with an active and reactive F (fluorine) element, making it easier to rebound with metals. An increase in its effects leads to a higher catalyst activity [1-5]. However, when it is added in surplus, it decreases the catalyst activity. This has been proven by Wen *et al.* [6] during research by adding KF in CaO, where its addition above 25%, decreased the catalyst activity. When the amount is large, it covers the surface of the catalyst, thereby reducing its activity. Hu *et al.* [7] also conducted a study of the addition of KF in several catalysts, such as CaO-Fe₃O₄, SrO-Fe₃O₄, and MgO-Fe₃O₄ in which each had the optimum condition with the addition of KF. This was shown from the acquisition of biodiesel. According to Hu *et al.* [7], the obtained biodiesel was high assuming the addition of KF reaches 25% for CaO, 35% for MgO, and 10% for SrO.

The dispersion of active metals to the surface of the solid material is capable of expanding the catalyst surface and increasing the number of

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Bulletin of Chemical Reaction Engineering & Catalysis, 14 (3) 2019, 478-489

Research Article

Pd-Fe₃O₄/RGO: a Highly Active and Magnetically Recyclable Catalyst for Suzuki Cross Coupling Reaction using a Microfluidic Flow Reactor

Hany A. Elazab^{1*}, Ali R. Siamaki², B. Frank Gupton^{2,3}, M. Samy El-Shall^{2,3}

¹Chemical Engineering Department, The British University in Egypt, BUE, Cairo, Egypt ²Chemical Engineering Department, Virginia Commonwealth University, Richmond, VA, United States of America, ³Chemistry Department, Virginia Commonwealth University, Richmond, VA, United States of America

> Received: 1st November 2018; Revised: 8th March 2019; Accepted: 13rd March 2019; Available online: 30th September 2019; Published regularly: December 2019

Abstract

There are several crucial issues that need to be addressed in the field of applied catalysis. These issues are not only related to harmful environmental impact but also include process safety concerns, mass and heat transfer limitations, selectivity, high pressure, optimizing reaction conditions, scale-up issues, reproducibility, process reliability, and catalyst deactivation and recovery. Many of these issues could be solved by adopting the concept of micro-reaction technology and flow chemistry in the applied catalysis field. A microwave assisted reduction technique has been used to prepare well dispersed, highly active Pd/Fe₃O₄ nanoparticles supported on reduced graphene oxide nanosheets (Pd-Fe₃O₄/RGO), which act as a unique catalyst for Suzuki cross coupling reactions due to the uniform dispersion of palladium nanoparticles throughout the surface of the magnetite - RGO support. The Pd-Fe₃O₄/RGO nanoparticles have been shown to exhibit extremely high catalytic activity for Suzuki cross coupling reactions under both batch and continuous reaction conditions. This paper reported a reliable method for Suzuki cross-coupling reaction of 4-bromobenzaldehyde using magnetically recyclable Pd/Fe₃O₄ nanoparticles supported on RGO nanosheets in a microfluidic-based high throughput flow reactor. Organic synthesis can be performed under high pressure and temperature by using a stainless steel micro tubular flow reactor under continuous flow reaction conditions. Optimizing the reaction conditions was performed via changing several parameters including temperature, pressure, and flow rate. Generally, a scalable flow technique by optimizing the reaction parameters under hightemperature and continuous reaction conditions could be successfully developed. Copyright © 2019 BCREC Group. All rights reserved

Keywords: Suzuki cross-coupling; 4-bromobenzaldehyde; Pd-Fe₃O₄/RGO; Flow reactor

How to Cite: Elazab, H.A., Siamaki, A.R., Gupton, B.F., El-Shall, M.S. (2019). Pd-Fe₃O₄/RGO: a Highly Active and Magnetically Recyclable Catalyst for Suzuki Cross Coupling Reaction using a Microfluidic Flow Reactor. *Bulletin of Chemical Reaction Engineering & Catalysis*, 14(3): 478-489 (doi:10.9767/bcrec.14.3.3518.478-489)

Permalink/DOI: https://doi.org/10.9767/bcrec.14.3.3518.478-489

1. Introduction

Over the past few decades, micro-reaction technology has been emerged as an ideal route

* Corresponding Author. E-mail: elazabha@vcu.edu (H.A. Elazab); to solve several critical issues in many aspects including organic chemistry and applied catalysis [1-8]. This new technology has created new promising horizons for chemical synthesis and industry via performing chemistry under continuous flow reaction conditions instead of the conventional batch chemistry [9-16]. This





Bulletin of Chemical Reaction Engineering & Catalysis, 14 (3) 2019, 490-501

Research Article

Polyvinylpyrrolidone - Reduced Graphene Oxide - Pd Nanoparticles as An Efficient Nanocomposite for Catalysis Applications in Cross-Coupling Reactions

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Received: 23rd October 2018; Revised: 14th March 2019; Accepted: 20th March 2019; Available online: 30th September 2019; Published regularly: December 2019

Abstract

This paper reported a scientific approach adopting microwave-assisted synthesis as a synthetic route for preparing highly active palladium nanoparticles stabilized by polyvinylpyrrolidone (Pd/PVP) and supported on reduced Graphene oxide (rGO) as a highly active catalyst used for Suzuki, Heck, and Sonogashira cross coupling reactions with remarkable turnover number (6500) and turnover frequency of 78000 h⁻¹. Pd/PVP nanoparticles supported on reduced Graphene oxide nanosheets (Pd-PVP/rGO) showed an outstanding performance through high catalytic activity towards cross coupling reactions. A simple, reproducible, and reliable method was used to prepare this efficient catalyst using microwave irradiation synthetic conditions. The synthesis approach requires simultaneous reduction of palladium and in the presence of Gaphene oxide (GO) nanosheets using ethylene glycol as a solvent and also as a strong reducing agent. The highly active and recyclable catalyst has so many advantages including the use of mild reaction conditions, short reaction times in an environmentally benign solvent system. Moreover, the prepared catalyst could be recycled for up to five times with nearly the same high catalytic activity. Furthermore, the high catalytic activity and recyclability of the prepared catalyst are due to the strong catalyst-support interaction. The defect sites in the reduced Graphene oxide (rGO) act as nucleation centers that enable anchoring of both Pd/PVP nanoparticles and hence, minimize the possibility of agglomeration which leads to a severe decrease in the catalytic activity. Copyright © 2019 BCREC Group. All rights reserved

Keywords: Graphene; Cross-Coupling; Microwave-assisted synthesis; Heterogeneous catalysis; Catalyst recycling

How to Cite: Elazab, H.A., El-Idreesy, T.T. (2019). Polyvinylpyrrolidone - Reduced Graphene Oxide - Pd Nanoparticles as an Efficient Nanocomposite for Catalysis Applications in Cross-Coupling Reactions. *Bulletin of Chemical Reaction Engineering & Catalysis*, 14(3): 490-501 (doi:10.9767/bcrec.14.3.3461.490-501)

Permalink/DOI: https://doi.org/10.9767/bcrec.14.3.3461.490-501

1. Introduction

Over the past few decades, Nanoscience has emerged as a new promising interdisciplinary scientific field [1-4]. Nanoclusters have been investigated for many catalytic applications due to their large surface-to-volume ratio. Recently, one of the main interests that attracted researchers' attention is transition metals based materials, especially when using palladium nanoparticles [5-10]. It is well established that palladium-based catalysts have been widely used in homogeneous and heterogeneous catalysis due to their several outstanding properties that combine between those of single metal atoms and other bulk metals [11-16]. In order to design new compounds with tailored chemical and

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