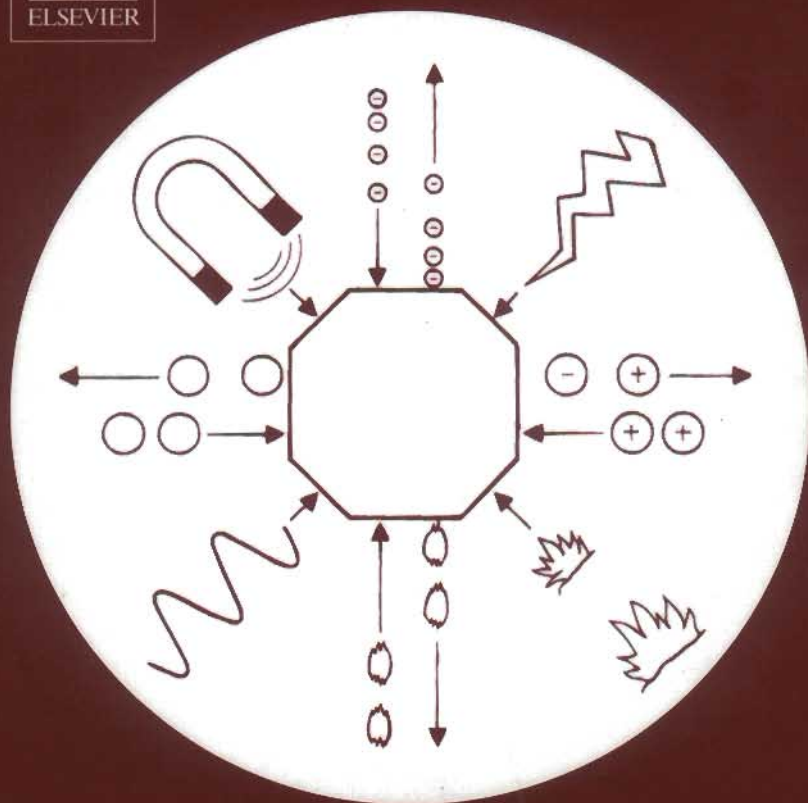


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**NEW DEVELOPMENTS AND
APPLICATION IN CHEMICAL
REACTION ENGINEERING**

**Hyun-Ku Rhee
In-Sik Nam
Jong Moon Park
(editors)**



Studies in Surface Science and Catalysis

Advisory Editors: B. Delmon and J.T. Yates

Series Editor: G. Centi

Vol. 159

NEW DEVELOPMENTS AND APPLICATION IN CHEMICAL REACTION ENGINEERING

**Proceedings of the 4th Asia-Pacific Chemical Reaction Engineering
Symposium (APCRE '05), Gyeongju, Korea, June 12-15, 2005**

Edited by

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First edition 2006

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Library of Congress Cataloging-in-Publication Data

A catalog record for this book is available from the Library of Congress

British Library Cataloguing in Publication Data

A catalogue record for this book is available from the British Library

ISBN-13: 978-0-444-51733-3

ISBN-10: 0-444-51733-2

ISSN (Series): 0167-2991

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Printed and bound in The Netherlands

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Preface

This Proceedings of APCRE'05 contains the articles that were presented at the 4th Asia-Pacific Chemical Reaction Engineering Symposium (APCRE'05), held at Gyeongju, Korea between June 12 and June 15, 2005, with a theme of "New Opportunities of Chemical Reaction Engineering in Asia-Pacific Region". The authors were invited to submit their manuscripts during the APCRE'05 at Gyeongju. It was understood that every manuscript should be reviewed by two experts in the corresponding area and, if accepted, the manuscripts will be published in the book series "Studies in Surface Science and Catalysis" by Elsevier BV early in 2006.

Following the tradition of APCRE Symposia and ISCRE, the scientific program encompassed a wide spectrum of topics, including not only the traditional areas but also the emerging fields of chemical reaction engineering into which the chemical reaction engineers have successfully spearheaded and made significant contributions in recent years. Indeed, APCRE'05 focused on the following areas and naturally the articles are classified into the same areas as in the APCRE'05:

- Biological and Biochemical Reaction Engineering
- Catalysis and Catalytic Reaction Engineering
- Chemical Reaction Engineering in Microelectronics
- Environmental Reaction Engineering
- Fluidized Bed and Multiphase Reactors
- Fuel Cells and Electrochemical Reaction Engineering
- Micro-reaction Technology
- Modeling, Simulation and Control of Chemical Reaction Systems
- Nano Materials Synthesis and Application
- Novel Reactors and Processes
- Polymer Reaction Engineering

Out of 284 papers presented at the APCRE'05, 190 papers have been accepted after a cautious review process and included in this Volume. In addition, six plenary lectures and 11 invited lectures are placed in two separate chapters in the front. One

author was allowed to have no more than four articles in the Proceedings. The articles were contributed by chemical reaction engineers and scholars from academia and R&D sector as well as from industrial sector, representing 16 countries not only from the Asia-Pacific region but from the western hemisphere.

We would like to express our sincere gratitude to all the authors for their valuable contribution and to the members of the Organizing Committee for sparing their valuable time and efforts to carry out the review process so successfully. We hope that this Proceedings may serve as noticeable references for the scientific and industrial communities in the years to come, contribute to make an overview of new developments and application in chemical reaction engineering, and allow chemical reaction engineering to make its full contribution to further advancement of respective countries in the Asia-Pacific region and other parts of the world.

Hyun-Ku Rhee
In-Sik Nam
Jong Moon Park

December 20, 2005

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Selective Conversion of Methane to C₂ Hydrocarbons using Carbon Dioxide as an Oxidant over CaO-MnO/CeO₂ Catalyst

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ABSTRACT

Carbon dioxide rather than oxygen seemed to be an alternative oxidant for the catalytic reaction of methane to produce C₂ hydrocarbons via oxidative coupling of methane (CO₂ OCM). The proper amount of medium and strong basic sites and the reducibility of the catalyst enhanced the CH₄ conversion and C₂ hydrocarbon yield, which may be due to the synergistic effect among CeO₂, CaO and MnO in the catalyst. The C₂ hydrocarbons selectivity and yield of 75.6% and 3.9%, respectively were achieved over the 12.8CaO-6.4MnO/CeO₂ catalyst. The catalyst showed a good stability for 20 h time on stream in the CO₂ OCM process.

1. INTRODUCTION

Direct conversion of methane to ethane and ethylene (C₂ hydrocarbons) has a large implication towards the utilization of natural gas in the gas-based petrochemical and liquid fuels industries [1]. CO₂ OCM process provides an alternative route to produce useful chemicals and materials where the process utilizes CO₂ as the feedstock in an environmentally-benefiting chemical process. Carbon dioxide rather than oxygen seems to be an alternative oxidant as methyl radicals are induced in the presence of oxygen. Basicity, reducibility, and ability of catalyst to form oxygen vacancies are some of the physico-chemical criteria that are essential in designing a suitable catalyst for the CO₂ OCM process [2]. The synergism between catalyst reducibility and basicity was reported to play an important role in the activation of the carbon dioxide and methane reaction [2].

In this paper, the selective conversion of methane to C₂ hydrocarbons over ternary CaO-MnO/CeO₂ catalysts in the CO₂ OCM process are presented. The synergistic effect between catalyst reducibility and distribution of basic sites are highlighted. The most promising catalyst was then tested towards its stability.

2. EXPERIMENTAL

The first ternary metal oxide catalyst of CaO-MnO/CeO₂ was prepared by simultaneous impregnation method, while the second ternary metal oxide of CaO/MnO-CeO₂ catalyst was prepared by combination of co-precipitation and impregnation method. The catalysts composition used in this paper were based on multi-responses optimization result [3]. H₂-TPR was carried out using Micromeritics 2900 TPD/TPR equipped by TCD. A catalyst amount of

about 0.05 g was purged with Ar ($25 \text{ cm}^3 \text{ min}^{-1}$) at 773 K for 1 h and was cooled down to room temperature. The flow of 6% H_2 in Ar ($25 \text{ cm}^3 \text{ min}^{-1}$) was then switched into the system, and the sample was heated up to 1223 K from room temperature at a rate of 5 K min^{-1} . CO_2 -TPD was carried out using Micromeritics 2900 TPD/TPR equipped by TCD. The catalyst samples of about 0.05 g each were initially calcined at 1073 K in a flow of argon ($25 \text{ cm}^3 \text{ min}^{-1}$) for 1 h. The chemisorption of CO_2 was carried out at 373 K by flowing CO_2 ($25 \text{ cm}^3 \text{ min}^{-1}$) for 1 h. The excess of CO_2 was purged in a flow of Ar ($25 \text{ cm}^3 \text{ min}^{-1}$) for 1 h. The sample was then heated to 1223 K at a linear heating rate of 5 K min^{-1} in a flow of Ar ($25 \text{ cm}^3 \text{ min}^{-1}$). The amount of H_2 uptake and the amount of CO_2 desorbed in both characterizations were detected using TCD. The performances of the catalysts were tested using a fixed-bed quartz reactor at the following conditions: reactor temperature = 1123 K; $\text{CH}_4/\text{CO}_2 = 1/2$, feed flow rate = 100 ml min^{-1} ; catalyst loading = 2 g. Before reaction, the catalyst was recalcined at 1123 K in air flow for 1 h and was flushed with high purity nitrogen at 1123 K for another 1 h. The products and the unreacted gases were analyzed by an online GC equipped with a thermal conductivity detector and PORAPAK N packed-column.

3. RESULTS AND DISCUSSION

3.1. Catalyst Characterization

The H_2 -TPR peaks of CeO_2 -based catalysts are presented in Fig. 1. The reduction of CeO_2 appears at about 300°C and two broad peaks are observed at about 540°C (weaker intensity) and 770°C [4]. The TPR peak at 540°C is assigned to the reduction of surface-capping oxygen of ceria [4], while the peak at 770°C can be ascribed to the reduction of bulk oxygen of CeO_2 . This reduction is associated to an increased reducibility of the bulk mixed oxide, which is evident in Figs. 1(c) and 1(d) as the H_2 -TPR peaks shift from 770°C to 760°C and 740°C for the CaO/CeO_2 and $\text{CaO-MnO}/\text{CeO}_2$ catalysts, respectively. The introduction of manganese strongly modifies the reduction behavior of CeO_2 by shifting the main peak of H_2 consumption to a lower temperature [5]. The TPR peak shifts show that the reducibility of both catalysts is different. The promotion of Ce^{3+} reduction is related to higher mobility of oxygen from chemisorbed CO_2 due to the introduction of manganese. The reduction of surface capping oxygen of $\text{CaO-MnO}/\text{CeO}_2$ catalyst was shifted to higher temperature (from 540 to 580°C) due to strong interaction effect of MnO_x and CeO_2 support. The interaction also generates a new strong H_2 -TPR peak at about 510°C which is attributed to reduction of Mn_2O_3 or MnO_2 to Mn_3O_4 . Meanwhile, the new small peak is also appeared at about 396°C due to the interaction effect. The H_2 -TPR spectra as depicted in Fig. 1 reveal that the $\text{CaO-MnO}/\text{CeO}_2$ catalyst has moderate reducibility with high medium and strong basic sites (CO_2 TPD). It is shown that the $\text{CaO}/\text{MnO-CeO}_2$ catalyst has higher MnO_x content than the $\text{CaO-MnO}/\text{CeO}_2$ catalyst as indicated by high intensity of TPR peak at 390°C . The very small peak within the temperature range of 380 - 400°C indicates that the catalyst synthesis method brings about a little proportion of MnO_2 form in the surface of $\text{CaO-MnO}/\text{CeO}_2$ catalyst which may be due to formation of solid solution of $\text{Ca}_{1-x}\text{Mn}_x\text{O}$. The strong H_2 -TPR peak at about 510°C is assigned to the reduction of Mn_2O_3 or MnO_2 to Mn_3O_4 , while the peak at about 580°C is attributed to final reduction from Mn_3O_4 to MnO . The color change of the catalyst from black to brown and/or grey during the reaction is in agreement with the results of H_2 -TPR peaks [6].

The CO_2 -TPD curves demonstrating the base strength distribution of different CeO_2 -based catalysts are presented in Fig. 2. From the figure, the difference in the distribution of basic sites for each catalyst indicates that the basicity and base strength distributions are significantly influenced by CaO and its interaction with MnO_x in the CeO_2 -based catalysts. The

CO₂ TPD spectra obviously exhibit that the CaO/CeO₂ catalyst gives the largest number of very strong basic sites, followed by CaO/MnO-CeO₂ and CaO-MnO/CeO₂ catalysts. According to Figs. 2(b-d), the doping CaO on the CeO₂-based catalysts results in the creation of a large number of medium, strong and very strong basic sites at the expense of the weak basic sites as compared to the pure CeO₂ catalyst [7]. The CO₂-TPD spectra in Figs. 2(c) and 2(d) show different peaks owing to the total number of medium and strong basic sites. Impregnation of calcium and manganese nitrate solutions to the CeO₂ catalyst leads to higher distribution of medium and strong basic sites rather than when the MnO-CeO₂ solid solution is used as the support. The CO₂-TPD peak of medium basic sites becomes more intense significantly for CaO/CeO₂ and CaO-MnO/CeO₂ catalysts, but not for CaO/MnO-CeO₂ catalyst.

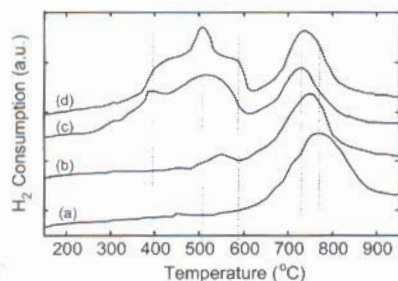


Fig. 1. H₂-TPD spectra of different catalysts. (a) CeO₂; (b) 12.8CaO/CeO₂; (c) 12.8CaO/6.4MnO-CeO₂; (d) 12.8CaO-6.4MnO/CeO₂

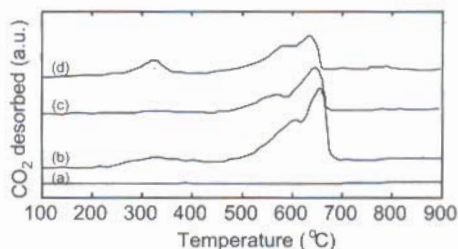


Fig. 2. CO₂-TPD spectra of different catalysts. (a) CeO₂; (b) 12.8CaO/CeO₂; (c) 12.8CaO/6.4MnO-CeO₂; (d) 12.8CaO-6.4MnO/CeO₂

3.2. Catalyst Activity and Correlation with Catalyst Basicity and Reducibility

The screening results of CeO₂-based catalysts over binary and ternary metal oxides in Table 1 indicate that the 12.8CaO-6.4MnO/CeO₂ catalyst is the most potential [3,8] with CH₄ conversion, C₂ selectivity and yield being 5.1%, 75.6%, and 3.9%, respectively. Addition of CaO to the pure CeO₂ catalyst results in a significant increase in the C₂ hydrocarbon selectivity. The enhancement in the C₂ yield is possibly due to the synergistic effect of MnO and CeO₂ where the reducibility of the catalyst are increased as revealed in the H₂-TPR results (Fig. 1). Our present study indicates that there exists a correlation between basic sites distribution, catalyst reducibility and catalytic activity toward C₂ hydrocarbons production as exposed in CO₂-TPD result (Fig. 2). The CaO species is suggested to play an important role in CO₂ chemisorption on the catalyst surface due to the role in distribution of medium and strong basic sites of the catalyst [9,10]. Proper amount of catalyst basicity, particularly medium and strong basic sites, greatly enhances the selectivity to C₂ hydrocarbons [2,9]. The MnO species evidently increases the reducibility of CeO₂ due to increasing oxygen mobility of the CeO₂ catalyst which enhances its reducibility and produces more oxygen vacancies [2]. The catalyst also shows high stability during 20 h time on stream for CO₂ OCM as revealed in Fig. 3.

Previously, we performed single- and multi-response optimization works in order to address optimal catalyst composition (%CaO and %MnO) and optimal operating conditions (temperature and CO₂/CH₄ feed ratio [3]. The maximum C₂ selectivity and yield of 76.6% and 3.7%, respectively were achieved in multi-responses optimization over the 12.8% CaO-6.4% MnO/CeO₂ catalyst corresponding to the optimum reactor temperature being 1127 K and CO₂/CH₄ ratio being 2 [3]. The recent contribution on the catalyst technology of CO₂ OCM was

developed by He *et al.* [11] using nano-CeO₂/ZnO catalyst achieved C₂ yield 4.8% and selectivity 83.6% at 1098 K. Due to still low yield in CO₂ OCM, further improvements are required including the exploitation of some non-conventional technologies.

Table 1. Catalysts performance results of CeO₂-based catalysts [3,8]

Catalysts	CH ₄ conversion (%)	C ₂ selectivity (%)	C ₂ yield (%)
CeO ₂	13.2	9.1	1.2
12.8CaO/CeO ₂	2.7	75.0	2.0
6.4MnO/CeO ₂	8.8	3.1	0.3
12.8CaO/6.4MnO-CeO ₂	5.3	62.2	3.3
12.8CaO-6.4MnO/CeO ₂	5.1	75.6	3.9

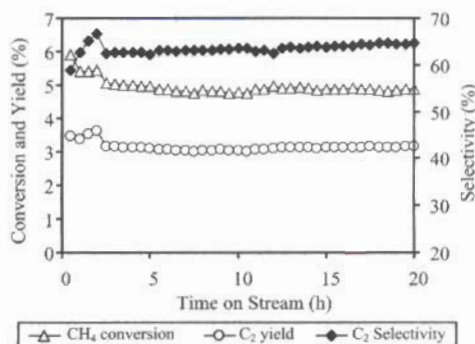


Fig.3. Stability test of 12.8CaO-6.4MnO/CeO₂ catalyst in CO₂ OCM reaction

4. CONCLUSIONS

Addition of CaO and MnO to the CeO₂ catalyst increased the CH₄ conversion and C₂ hydrocarbons yield in CO₂ OCM. The enhancement may be due to the synergistic effects between highly number of medium and strong basic sites and high reducibility of catalyst. The 12.8CaO-6.4MnO/CeO₂ catalyst exhibited better performance and high stability for the CO₂ OCM process.

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