MODIFICATION OF ZEOLITE HZSM-5 FOR METHANE CONVERSION TO HIGHER HYDROCARBONS

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To my parents, beloved wife and daughters……

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ABSTRACT

The direct conversion of methane to liquid hydrocarbon has not yet been successfully economized in an inexpensive process. Acidic zeolites were shown to have reasonable activities for the formation of higher hydrocarbons from methane, however the selectivity was low. The presence of acid sites leads to the oxidation of the higher hydrocarbons formed. Boron incorporation on zeolite would reduce the acidity and thus improved the selectivity. The purpose of this study is to modify the ZSM-5 zeolite with boron and impregnate them with metal for methane conversion. The catalysts were characterized for their structure and acidity. The conversion of methane in the presence of oxygen using packed bed micro reactor has been investigated. Direct substitution of boron for aluminium in the HZSM-5 zeolite structure (BZSM-5), resulted in slightly lower methane conversion (23% compared to 35%) due to its lower acidity, however it improved the higher hydrocarbon selectivity. The introduction of copper species in the catalyst (Cu1%BZSM-5) improved the methane conversion (51%) and the higher hydrocarbon selectivity (11.5%). The effects of operating conditions such as temperature, oxygen concentration, feed flow rate and amount of copper loading on the activity of the catalyst were studied. To examine the importance of further oxidation of intermediate product, the reaction between ethylene and oxygen was carried out using the similar operating conditions with that of methane. The result showed that Cu1%BZSM-5 was capable to convert ethylene to higher hydrocarbons even though it has low acidity. Ethylene conversion and higher hydrocarbon selectivity achieved were 86% and 87% respectively while carbon oxides selectivity was 13%.

ABSTRAK

Proses penukaran metana secara terus kepada hidrokarbon cecair sehingga kini masih belum berjaya mencapai tahap yang ekonomi yang berpatutan. Zeolit yang berasid menunjukkan aktiviti yang munasabah untuk pembentukan hidrokarbon yang lebih panjang daripada metana, walaupun begitu tahap kememilihannya masih rendah. Kehadiran tapak berasid menyebabkan pengoksidaan hidrokarbon panjang yang terbentuk. Penambahan boron ke dalam zeolit dijangkakan akan merendahkan keasidan dan seterusnya memperbaiki kememilihan. Tujuan kajian ini ialah untuk memodifikasikan zeolit ZSM-5 dengan boron dan mengisi tepukan dengan logam untuk tindak balas penukaran metana. Pencirian mangkin tersebut akan dilakukan berdasarkan struktur dan keasidan. Tindak balas penukaran metana dengan oksigen akan dilakukan menggunakan reaktor mikro lapisan terpadat. Penggantian terus aluminium dengan boron di dalam struktur HZSM-5 zeolit (BZSM-5), menyebabkan sedikit penurunan dalam penukaran metana (23% dibandingkan dengan 35%) disebabkan oleh penurunan keasidan, walau bagaimanapun terdapat peningkatan kememilihan hidrokarbon panjang. Kehadiran spesis kuprum di dalam mangkin (Cu1%BZSM-5) telah meningkatkan penukaran metana (51%) dan kememilihan hidrokarbon panjang (11.5%). Kajian kesan keadaan operasi yang berbeza seperti suhu tindakbalas, kepekatan oksigen, kadar alir masukan dan juga jumlah kuprum di dalam mangkin, ke atas aktiviti mangkin juga dijalankan. Untuk menilai kepentingan pengoksidaan hasil perantaraan, tindak balas etilena dengan oksigen dijalankan dengan keadaan operasi yang sama seperti tindak balas metana. Keputusan menunjukkan Cu1%BZSM-5 mampu untuk menukarkan etilina kepada hidrokarbon panjang walaupun mempunyai keasidan yang rendah. Penukaran etilina dan kememilihan hidrokarbon panjang yang diperolehi ialah 86% dan 87% masing-masing sementara kememilihan karbon oksida ialah 13% sahaja.

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NOMENCLATURES

2θ	Degree of Diffraction
λ	The wavelength of the beam
a.u.	Arbitrary unit
C_2^+	Higher hydrocarbon with carbon number equal to two and above but below five, such as ethane, ethylene, propane and butane.
C_5^+	Higher hydrocarbon with carbon number equal to five and above such as pentane, hexane,and pentene.
CO_x	Carbon dioxide and carbon monoxide
FID	Flame Ionization Detector
FTIR	Fourier Transform Infrared Spectra
GC	Gas Chromatography
IR	Infrared
IR-Py	Infrared Pyridine
MTBE	methy tertiary-butyl ether
NH_3-TPD	Ammonia- Temperature Programmed Desorption
OCM	Oxidative Coupling of Methane
TPD	Temperature Programmed Desorption
UV	Ultra violet spectroscopy
W/F	Ratio of catalyst weight to feed flow rate
XRD	X-Ray Diffraction
ZSM-5	Zeolite Socony Mobil Number 5

CHAPTER 1

INTRODUCTION

Malaysia has large, uncommitted reserves of natural gas. Natural gas has currently been made ready for use either as ordinary gas in pipelines or as physically liquefied gas shipped on tankers. Liquefied gas is really the only alternative method viable when the reserves are located far from the major customers. A constraint however on the growth of liquefied gas has always been the slow build up of the liquefied gas and the construction of regasification capacity (Heng and Idrus, 2004). Traditionally, there are two competing demands for natural gas. Firstly, as a clean fuel in power generation, industrial kilns, furnaces and domestic heating and secondly, as feedstock for petrochemical industries. The utilization of natural gas is limited by difficulties in transportation. Transportation of gas over long distances is energy and capital intensive. Therefore, much of the remote gas, co-produced with oil, is either flared or reinjected into the reservoir. It would be desirable to convert methane on site to gasoline range hydrocarbons and then transport the high-value fuel to worldwide markets.

The consumption of transportation fuels is increasing continuously, while the reserves of crude oil are declining. The extra supplement of fuels is needed due to the predicted shortfall in the world crude oil production (Abosedra *et al.*, 2004). There are a lot of natural gas around the world and thus, it is an incentive for converting methane into transportation fuels or higher hydrocarbons.

During the last decade considerable effort has been devoted to catalytic conversion of methane to higher hydrocarbons. Methane activation is observed to be a quite slow process except at high temperature and the lack of reactivity is due to the stability of the bonds linking the carbons to hydrogen (Lunsford, 1990; Han *et al.*, 1994).

The conversion of natural gas to liquid hydrocarbon has not yet been successfully economized in an inexpensive process. In general, there are two types of routes for methane conversion to hydrocarbon: indirect and direct methods. The indirect route for methane conversion (e.g. Fischer-Tropsch, Mobil Process and Shell Middle Distillate Synthesis) requires, as the first step, the production of synthesis gas (CO and H_2) from methane by costly and inefficient process of steam reforming. The direct route of methane conversions to higher hydrocarbons has attracted academic and industrial interests due to its potential to be effective for utilizing natural gas as industrial feedstock. However, the usefulness of this process has been limited so far as it has not been possible to achieve large scale conversion of methane directly to higher hydrocarbons.

1.1 Problem statement

Recently, new processes for direct methane conversion to hydrocarbon are reported. One of the processes is called Partial Oxidation Process where methanol is produced (Krylov, 1993; Herman *et al.*, 1997; Raja *et al.*, 1997; Zhang *et al.*, 2002; Michalkiewicz, 2004). In this process methane reacts with oxygen in the presence of catalyst to produce methanol according to the following reaction;

$$CH_4 + 1/2\ O_2 \longrightarrow CH_3OH$$

Depending on the catalyst, the process temperature ranges from 700 up to 900°C, and the pressure being close to atmospheric. In most cases, mixed oxide catalyst is used as a catalyst (Herman *et al.*, 1997). Unfortunately, the yield of methanol is too low (below 10%) and beyond industrial interest (Zhang *et al.*, 2002; Michalkiewicz, 2004, Michalkiewicz, 2006).

Another method for direct conversion of methane to hydrocarbon is called Oxidative Coupling Process (Yoon *et al.*, 1997; Shischak *et al.*, 1997; Choudary *et al.*, 1998; Kus *et al.*, 2003; Huggil *et al.*, 2005). Oxidative coupling of methane to produce C_2^+ hydrocarbons achieved a large interest in the last few years. The immense research effort has led to a significant increase in the activity and selectivity of the applied catalysts. However, further increase in C_2^+ selectivity and yields by catalyst improvement or by reaction engineering means it is necessary to make the process commercially viable. So far, the maximum yield obtained is about 20%, which means that the process is economically unfeasible (Huggil *et al.*, 2005)

Zeolite based catalysts are successfully employed in a variety of commercial processes in the petroleum and petrochemical industries (Szostak, 1989). Despite this fact, there are only a few investigations on the potentials and usefulness of modified zeolites as catalysts for the formation of higher hydrocarbons from methane. Stimulated through the observation of polycondensation of methane in liquid super acids by Olah *et al.* (1968), strong acidic zeolites have been tested for their ability to catalyze the formation of higher hydrocarbons (Shepelev *et al.*, 1983; Anderson, 1985; Kowalak *et al.*, 1988; Weckhusyen *et al.*, 1998; Xu and Lin, 1999;Yuan *et al.*, 1999; Liu *et al.*, 2004; Burns *et al.*, 2006). Direct conversion of methane to mainly ethane and ethene has been observed over fluorinated H-mordenite zeolite at 525^{o}C, but with very poor methane conversion far below 1% (Kowalak *et al.*, 1988). These low conversions are quite expected due to an unfavorable thermodynamic equilibrium at temperatures below 1000^{o}C.

The thermodynamic limitations for methane conversion can be overcome by adding an oxidizing reagent in the feed. Shepelev *et al.* (1983) and Anderson *et al.* (1985) studied the conversion of methane in the presence of N_2O and O_2. Oxidation with nitrous oxide over HY and HZSM-5-type zeolites resulted in a higher yield of hydrocarbons as compared to that found with oxygen. With the latter oxidant, carbon oxides were produced almost exclusively. From an economic point of view, however, the use of N_2O for methane conversion is too expensive. Hence, the more recent investigations using zeolites or zeolite-like materials as catalysts focus on the use of oxygen or air as oxidizing agents. From the experimental data (Shepelev *et al.*,1983; Anderson, 1985; Garnet,1988), it can be concluded that (i) strongly acidic zeolites in the presence of oxygen catalyze the complete oxidation of methane to CO_x and H_2O and (ii) the catalyst for the desired formation of higher hydrocarbons from methane/oxygen mixtures can be prepared by the modification of non-acidic zeolites with certain oxides.

Direct partial oxidation of methane with O_2 to higher hydrocarbons and in particular C_5^+ liquid over transition metal containing ZSM-5 catalyst is reported (Han *et al.*, 1994a; Nor Aishah and Anggorro, 2004). Han *et al.* (1994a) found that the successful methane conversion to hydrocarbons over metal-containing ZSM-5 catalyst is due to two effects: (i) ability of the metal to show some limited activity to generate olefins from methane, and (ii) sufficient low olefin oxidation activity to allow the olefin produced to remain in the system. In another study by Han *et al.* (1994b), it was found that Zn loaded on ZSM-5 beyond one wt% metal potentially reduced desired catalyst acidity needed for the conversion of methanol produced to C_5^+. Nor Aishah and Anggorro (2004) studied the direct conversion of methane to higher hydrocarbons using ZSM-5 modified with transition metal. They claimed that the successful production of higher hydrocarbons from methane depends on the amount of aluminium in the zeolite framework and the strength of the Brönsted acid sites. The low hydrocarbon yields demonstrated that the modification was performed at the expense of the oxidation and acidic features of the ZSM-5.

Methane dehydroaromatization in the absence of gas-phase oxygen has received considerable recent attention as a promising route for direct conversion of methane into highly value-added chemicals (Weckhusyen *et al.*, 1998; Liu and Xu, 1999; Wu *et al.*, 2005; Burns *et al.*, 2006) Up to now, most research work is focused on Mo/HZSM-5 catalysts, prepared by either the impregnation or the solid-state reaction method. It is generally accepted that both the activation of the methane C-H bond and the formation of the initial C-C bond occur on the reduced Mo carbide species, which is formed from the reduction of MoO_x species by CH_4 in the early stage of the reaction, whereas the oligomerization, cyclization, and aromatization of the C_2 hydrocarbon species are catalyzed by the Brönsted acid sites of the HZSM-5 zeolite.

The conversion of methane to benzene over transition metal ion ZSM-5 zeolite without an oxidant has been carried out by many researchers (Weckhusyen *et al.*, 1998; Wang *et al.*, 2004; Liu *et al.*, 2004; Burns *et al.*, 2006). Weckhusyen *et al.* (1998) claimed that a benzene selectivity of about 30% at a CH_4 conversion of 3.2% was achieved. The effects of preparation and the metal loading were studied for the methane conversion. It is evident that the catalytic properties depend on the presence of Brönsted acid sites and the distribution of the transition metal ions in the zeolite material. For catalyst prepared by solid-state ion-exchanged zeolites, the metal ions were predominantly located in the zeolite channels. In contrast, for the catalysts prepared by impregnation, the transition metal ions were at the outer surface of the zeolite. The solid-state ion-exchange zeolite has lower Brönsted acid sites and thus gives lower methane conversion. Furthermore, they revealed that higher aromatic compounds, such as naphthalene cannot be formed by this catalyst since the zeolite channels were partially blocked by metal ions.

The shape selectivity and acidity of zeolite ZSM-5 has generated great interest. Several attempts to modify the properties of zeolite ZSM-5 have been pursued by incorporating elements of different sizes and different chemical features (Howden, 1985; Chu *et al.*, 1985b; Han *et al.*, 1992; Derewinski *et al.*, 1994; Lu *et al.*, 2003; Nor Aishah and Anggorro, 2004). Ratnasamy *et al.* (1986) and Derewinski (1994) compared the acidity and catalytic activity of aluminosilicate and borosilicate ZSM-5 zeolites. They found that the acidity of borosilicate ZSM-5 was lower than aluminosilicate ZSM-5 zeolite. In the study of methanol conversion to hydrocarbon, they reported that borosilicate zeolite produced more light olefins as compared to aluminosilicate due to the suppression of acid sites on the zeolite.

Ernst *et al.* (1989) used ZSM-5 zeolite catalyst for the methane conversion. The oxidation of methane with ZSM-5 produced carbon oxides due to strong zeolite acidity. The presence of transition metal on ZSM-5 zeolite generally increased the methane conversion (Han *et al.*, 1994). The transition metal oxide located on the surface increase the concentration of surface oxygen and hence oxidative reaction is facilitated (Larkins *et al.*, 1988). The low production of higher hydrocarbons, C_5^+, is expected due to higher acidity of ZSM-5 catalyst. Hinsen *et al.* (1984) reported that an acidic catalyst resulted in low selectivity to hydrocarbons and high selectivity to CO_x. By introducing boron into the zeolite ZSM-5, the acidity of zeolite is suppressed (Ratnasamy *et al.*, 1986; Derewinski, 1994; Lu *et al.*, 2003). Furthermore, Sofranco *et al.* (1988) claimed that methane conversion to higher hydrocarbons is possible using boron-promoted reducible metal oxide catalyst. Methane conversion to C_2^+ is improved when reducible metal oxide in the presence of boron is used as compared to reducible metal oxide only. The conversion of methane is 38% with 66% selectivity to C_2^+ hydrocarbons product. In this case, boron acts as a promoter. Abrevaya *et al.* (1990) claimed that boron compound is an essential catalytic component for methane conversion to C_2^+ hydrocarbons. The catalyst consisting essentially of a solid oxides of boron, tin and zinc having a composition expressed in terms of mol ratios of elemental zinc, tin and boron components of: 1.0 Zn: 0.1-5.0 Sn: 0.1-7.0 B.

It is expected that the conversion of methane to gasoline range hydrocarbon (C_5^+) is possible. Methane oxidative coupling to produce light olefins over a metal component and further reaction of the olefins produced over a zeolite catalyst could provide an alternative route to synthetic gasoline production. Zeolite has been a particularly important catalyst in gasoline production because

i) Its pore structure provides shape selectivity
ii) Its high acidity promotes oligomerisation, isomerisation, cracking and aromatisation reactions involved in the restructuring of the primary products
iii) Its resistance to coking and
iv) Its stability under high temperature conditions.

The methane oxidative coupling step is thought to be the first step in methane oxidation to produce olefin. Higher hydrocarbons (C_5^+) will be produced from reaction of olefin through oligomerization, dehydrocyclization and aromatization using zeolite base catalyst according to the reaction scheme below:

$$CH_4 + O_2(cat) \text{ -----> } \underset{CO_x,\ H_2}{C_2H_6} \text{ -----> } C_2H_4 \text{----->}$$

Oligomerization,
dehydrocyclization and
aromatization yielding
C_5^+ hydrocarbons

The combination of the oxidative catalysis of methane (i.e. transition metal) with acid catalyst (i.e. pentasil zeolite) in a single catalytic process over bifunctional oxidative-acid catalyst would be possible to convert methane directly to liquid hydrocarbon. This could be done by modifying pentasil zeolite catalyst with a suitable element. One possibility of modifying zeolites is the replacement of some of the silicon and aluminum with boron and transition metal. Boron is of great importance as a substitutional element for aluminium (Keading *et al.*, 1981; Holderich *et al.*, 1984). It was conceived that replacing Al with boron (B) in ZSM-5 would enhance the catalytic activity. Also because of its smaller atom, the incorporation of B in the lattice would reduce the unit cell volume into a more selective zeolite. The metal loaded on the catalyst will act as a bifunctional catalyst. The present study will examine the properties and activities of B-ZSM-5 containing various amounts of boron for methane conversion. The effect of the first row transition metal loading on the property and activity of B-ZSM-5 is also investigated. The first row transition metals were selected in the study because they have shown to produce higher hydrocarbon yields from direct partial oxidation of methane over ZSM-5 loaded with the first row transition metal (Han *et al.*, 1994; Anggoro 1999; Nor Aishah and Anggoro, 2003). Furthermore these metals are easily obtained as compared to other transition metals.

It is essential to have a thorough understanding of the relationship between the physico-chemical and its catalytic properties. The research questions and statements of the problem are shown in Figure 1.1.

1.2 Research Objectives

The objectives of this work are

1) To synthesize the H-ZSM-5 and Boron-ZSM-5 zeolite by direct synthesis.
2) To modify H-ZSM-5 with boron and the first row transition metal elements so that a bifunctional oxidative and acidic catalyst could be produced.
3) To investigate the physicochemical properties of the modified catalysts.
4) To test the activities of the modified catalyst for the methane conversion to higher hydrocarbon in a single catalytic process.
5) To study the effect of boron loading on catalyst properties and performance.
6) To investigate the effects of temperature, reactant ratio and reactant flow rate on methane conversion and higher hydrocarbons selectivity.
7) To investigate the stability of intermediate products of methane conversion and its reaction to higher hydrocarbons.

Research Questions

1. **What is the structure of the catalyst produced?**
2. **What are the physicochemical properties of the catalysts ?**
3. **How active are the catalysts for methane conversion?**
4. **How are the structure, physicochemical and catalytic properties interrelated?**

Research Problem

Are the ZSM-5 containing boron and selected first row transition metals potential catalysts for the conversion of methane to higher hydrocarbons ?

Figure 1.1: Schematic representation of the research questions and statement of the problem.

1.3 Scopes Of Work

The few economic evaluations available in the literature indicate that the feasibility of methane conversion to higher hydrocarbons largely depends on the catalyst performance; a methane conversion in the 38% range and hydrocarbons selectivity of 85% can render this process economically competitive (Korf, 1990; Zaman, 1999). Only by furthering our knowledge of the reaction mechanism and the role of the catalyst will it be possible to achieve this difficult but hopefully not impossible goal.

The development of the heterogeneous catalyst may be regarded as an interactive optimization process, basically consisting of three steps, namely preparation, characterization and testing of the catalyst. Therefore, the scopes of work in this study were divided into three stages: Preparation and modification of the catalysts; characterization of the catalysts for its physicochemical properties; and the reactivity of the catalysts for methane conversion to higher hydrocarbons. On the reactivity test, the effects of reaction temperature, feed flow rate and oxygen ratio were carried out.

1.4 Outline of the Dissertation

This dissertation is a report on a series of studies, systematically conducted to achieve the above objectives. It is divided into seven chapters. Chapter 2 presents the literature review on the methane conversion to higher hydrocarbon and application of zeolite on this reaction. Chapter 3 addresses the effect of boron loading on ZSM-5 zeolite and its physicochemical properties. In this chapter, the preparation and characterization of the catalyst are discussed. The effects of transition metal loading on the catalyst are also discussed. Chapter 4 presents the results on the methane conversion on this modified catalyst.

The study on the effects of operating conditions such as temperature, oxygen concentration and feed flow rate on the activity of the catalyst are discussed in Chapter 5. Ethylene, which is the primary product of methane activation, undergoes subsequent oligomerization on zeolites to form higher hydrocarbons or further oxidation to form carbon oxides. In order to examine the importance of further oxidation of intermediate product, the reaction of ethylene with oxygen was carried out. The results were discussed in Chapter 6. Chapter 7 concludes the findings of this study and recommends topics for future research.

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