

EFFECT OF LITHIUM CHLORIDE (LiCl) DOPANT ON THE PERFORMANCE OF CATALYSTS FOR THE OXIDATIVE COUPLING OF METHANE

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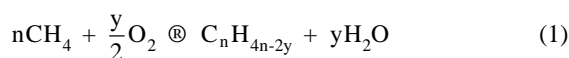
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Abstract: The present study reports effects of lithium chloride (LiCl) doping on MgO, La₂O₃, SnO₂, CaO and ZnO catalysts. All the catalysts were prepared by the impregnation method. The catalysts were tested at reaction temperature of 775°C. The feed flow rate of methane, oxygen and nitrogen was carried out in the ratio of 3:1:2 giving a weight hourly space velocity (WHSV) of 0.375 g.s/cm³. La₂O₃ showed the highest C₂₊ selectivity among the undoped catalysts. All the catalysts were doped with LiCl to compare their methane conversion, selectivity and product yield. The 2.0 mol% LiCl doped into La₂O₃ was the best catalyst formulation having achieved 46.7% of C₂₊ selectivity with CH₄ conversion of 29.0% and the increase in selectivity was attributed to the presence of chlorine. Hydrogen production was more pronounced for MgO based catalysts and believed to be generated by surface-catalyzed reactions.

Key Words: Oxidative Coupling, C₂₊ Selectivity, Hydrogen Production, Ethylene, Chlorine

1. INTRODUCTION

Direct conversion of methane to industrial organic compounds is particularly significant especially natural gas, where the major component is methane, is one of the most plentiful fossil fuels available. Industry analysts estimate that the world holds enough readily recoverable natural gas to produce 500 billion barrels of synthetic crude [1]. Perhaps triple that amount of gas can be found in coal seams, natural gas hydrates [2] and in formations that release the gas only slowly. Crude oil is now the main source of chemicals and liquid fuels. However, with the continual depletion of crude oil reserve, the demand for natural gas as a source of chemicals and fuels would certainly increase [3]. One route is the direct dimerization of methane through the catalytic oxidative coupling of methane (OCM) process. The catalytic oxidative coupling of methane (OCM) is represented by the following equation:



In most published works in this topic, n would have the optimum value of 2. Oxidant other than O₂, such as N₂O

and O₃ have also been shown to facilitate the coupling reaction [4].

The hydrocarbon products are often reported as C₂₊ products, mostly ethane and ethylene as the major components with ethylene as the target product. C₃ hydrocarbons are usually formed but at comparatively low selectivity. In addition to these products, CO_x (CO and CO₂) are also formed nonselectively. The conversion of CH₄ to CO_x is undesirable since it represents a lower yield of hydrocarbons. The CO_x may also poison the surface of the catalyst. Another important product from the reaction is hydrogen, which is also a valuable product especially in fuel cells, which is projected to become the main source of energy for automobiles [5].

A large number of compounds, mostly metal oxides has been tested and found capable of facilitating the oxidative coupling reaction. In most of the catalysts studied, reaction temperatures in the range of 700-900°C are needed and products consists of C₂₊ hydrocarbons, carbon monoxide, carbon dioxide and water are obtained. There appears to be an inverse relation between methane conversion and selectivity to C₂ hydrocarbons, resulting in an upper limit of selectivity per pass through the reactor. Indeed, investigation on the thermodynamic feasibility of the reactions, proposed that ethylene yield of 30 per cent is the highest that can be achieved [6].

In earlier studies, the goal of the applied research was often to maximize C₂₊ yield by varying composition of the catalyst, partial pressures of reactants, etc.; however, even in the best catalytic systems, the C₂₊ hydrocarbons concentration in the exit gas were quite low making it economically undesirable to extract C₂₊ from such a mixture.

The more effective catalysts may be divided into five groups a) highly basic pure oxides, of which the early members of the lanthanide oxides series (excluding CeO₂) are the best, b) Group IA or IIA ions supported on basic oxides (for example, Li/MgO, Ba/MgO and Sr/La₂O₃), c) monophasic oxides, d) a few transition metal oxides that contain Group IA ions, and e) any of these materials that are promoted with chloride ions.

It is very unlikely that only a single type of site is responsible for the activation of methane as can be seen in the diversity of the catalysts used in the oxidative coupling reaction. It is difficult to determine the nature of the active sites as most characterization methods are

applicable only at conditions far removed from those used in the actual catalytic reaction. Nonetheless, rational hypothesis can be made concerning the species that might exist on the surface of the functioning catalysts.

The addition of chloride ions to an oxidative coupling catalyst can have a marked effect on its properties, particularly with respect to the ethylene (C_2H_4) to ethane (C_2H_6) product ratio. The chloride may be introduced either initially as a part of the catalyst or through organo chlorine compounds, for instance CCl_4 , that are added to the reagents^[7]. Because chlorine is known to dehydrogenate C_2H_6 in the gas phase, it has been suspected that homogenous reactions may be responsible for the large C_2H_4/C_2H_6 ratios that are observed in these chlorine containing systems^[8]. In this study, the effects of LiCl on different oxide catalysts such as MgO, La_2O_3 , SnO_2 , CaO and ZnO are reported.

2. EXPERIMENTAL

Catalysts were prepared through the wet impregnation method. Powdered MgO (Merck, extra high purity), La_2O_3 (Fluka, purity 97%), CaO(BDH), ZnO (Merck, 99.0%) and SnO_2 (Merck, 99.0%) were used directly from supplier's package without any treatment. For doping the catalysts with LiCl, desired amount of LiCl (Merck, 98.0%) was dissolved in deionized water. The supports were then poured into the solution and stirred. The resulting paste was dried in an oven for 12 hrs at $110^\circ C$ - $120^\circ C$. The dried paste was then crushed to powder and calcined at $950^\circ C$ for 4 hour. The calcined material was then pelletized at 5 tons/ m^2 for each 5 gm of catalyst. The pellet was then calcined again at $950^\circ C$ for another 8 hrs. After calcination, the pellets were crushed and sieved to 40-60 mesh size. The same procedure was applied with the other dopant (Li_2CO_3 , Ajax, 99.5%) used before testing.

The catalysts were tested in a stainless steel microreactor (O.D. 12.7 mm, I.D. 10.92 mm and length 600 mm) situated vertically in a tubular furnace (Carbolite VST 12). The catalyst layer was placed in the center of the microreactor. The free space below and above the catalyst layer was filled with quartz particles (RDH) of 40-60 mesh size in order to minimize the dead volume of the reactor. Methane (Malaysian Oxygen, purity 99.99%), Oxygen (99.8%) and Nitrogen (99.99%) were passed through the microreactor. Flow of the gases was controlled using mass flow controllers (Brooks 5850E for both nitrogen and oxygen and MKS for methane). Outlet gas flow rate was monitored using a gas flowmeter (Alexander Wright DM3 B). The gaseous products were analyzed using an on-line gas chromatograph (Hewlett-Packard 6890). Poropak N column was used to separate carbon dioxide, ethane, ethylene and propylene and Molecular Sieve 5A was used for separation of hydrogen, oxygen, carbon monoxide, nitrogen and methane. Water, a by-product of the reaction, was trapped in a gas trap before gaseous product sampling was carried out. The gas chromatograph was calibrated using a standard gas mixture supplied by BOC Gases, U.K.

The catalyst was first heated in O_2 at a flow rate of 10 ml/min at $800^\circ C$ for half an hour to oxidize adsorbed components. It was then cooled down to $700^\circ C$ before mixture of reactants was fed through the microreactor with $CH_4:O_2:N_2$ ratio of 3:1:2 giving a total flow rate of 240 ml/min. A 1.5 gm of catalyst was used for each experimental run. Furnace temperature was adjusted to the desired reaction temperature. Catalyst bed temperature was monitored using a Chromel-Alumel thermocouple inserted into the catalyst bed. Once the bed temperature stabilized for 15 mins, the sample was drawn.

The activity of the catalysts was expressed in terms of methane conversion, selectivity and yield for C_{2+} hydrocarbon and hydrogen. A carbon balance of $100\pm 2\%$ was obtained for every run over the catalysts. The conversion of methane or oxygen was defined as

$$X(CH_4 \text{ or } O_2) = \frac{\text{moles } CH_4 \text{ or } O_2 \text{ converted}}{\text{moles } CH_4 \text{ or } O_2 \text{ fed}} \cdot 100\% \quad (2)$$

The selectivity for C_n products was calculated as

$$S(C_n) = \frac{n \cdot \text{moles } C_n \text{ in products}}{\text{moles } CH_4 \text{ converted to all products}} \cdot 100\% \quad (3)$$

The yield for C_n product was given by

$$Y(C_n) = \frac{n \cdot \text{moles } C_n \text{ in products}}{\text{moles } CH_4 \text{ fed}} \cdot 100\% \quad (4)$$

The selectivity for H_2 product was calculated as

$$S(H_2) = \frac{2 \cdot \text{moles } H_2 \text{ in products}}{4 \cdot \text{moles } CH_4 \text{ converted to all products}} \cdot 100\% \quad (5)$$

The yield for H_2 product was given by

$$Y(C_n) = \frac{2 \cdot \text{moles } H_2 \text{ in products}}{\text{moles } CH_4 \text{ fed}} \cdot 100\% \quad (6)$$

3. RESULTS

In most of experimental runs, methane to oxygen mole ratio was more than 2. A 100% O_2 conversion was achieved in most of experiments. Figure 1 shows the methane conversion, selectivity and yield of C_{2+} hydrocarbons for undoped catalysts. La_2O_3 gives the highest activity in terms of selectivity and yield of C_{2+} hydrocarbons, which were 44.8% and 12.4% respectively, followed by MgO with 39.4% selectivity and yield of 11.3% of C_{2+} hydrocarbons.

ZnO gave 18.8% selectivity OF C_{2+} hydrocarbons and yield of 4.0%. Catalytic performance of both SnO_2 and CaO show that they are nonselective catalysts. All these results are in line with literature findings. Both the La_2O_3 and MgO have been known to be good coupling catalysts especially when doped with alkaline earth oxides^[9].

Fig. 1 Performance of undoped catalyst

Figure 2 shows the selectivity and yield of hydrogen for the undoped catalysts. MgO showed the highest activity with H₂ selectivity of 19.1% and yield of 5.5%. Compared to the selectivity and yield of hydrocarbons product, CaO showed significant selectivity and yield of H₂ relative to MgO. A selectivity of 12.0% and 2.7% yield of hydrogen were obtained. Both SnO₂ and ZnO gave low activity to H₂ formation.

The ratios of C₂H₄/C₂H₆ and CO/CO₂ are shown in Figure 3. From these results, it appeared that higher C₂₊ selectivity leads to higher C₂H₄/C₂H₆ ratio, nevertheless a small degree of variation in catalytic activity among the catalysts was observed. MgO produced higher ethylene relative to ethane as compared to La₂O₃ even though La₂O₃ gave the highest selectivity to C₂₊ hydrocarbons. The C₂H₄/C₂H₆ ratio did not correlate with H₂ selectivity and yield.

Fig. 2 Activity of undoped catalyst for selectivity and yield of hydrogen production**Fig. 3** Ethylene to ethane ratio and carbon monoxide to carbon dioxide ratio over undoped catalyst

Doping the catalysts with 1 mol% LiCl did not result in an apparent increase in terms of C₂₊ selectivity and yield for any of the catalysts except CaO, as shown in Figure 4. The C₂₊ selectivity of 1 mol% LiCl/CaO increased much higher compared to the other catalysts but the methane conversion decreased. The C₂₊ selectivity increased from 7.6% to 17.2% while methane conversion dropped 22.8% to 18.8%. No marked changes in the measured parameters were observed for other 1 mol% LiCl doped catalysts.

Figure 5 shows the selectivity and yield of H₂ on the 1 mol% LiCl doped catalysts. Again, except for 1 mol% LiCl/CaO, there are no significant changes in both the H₂ selectivity and yield for the other catalysts. The H₂ selectivity and yield for 1 mol% LiCl/CaO catalysts decreased from 12.0% to 0.9% and from 2.7% to 0.2%, respectively.

Fig. 4 Activity of 1 mol% of LiCl doped catalysts

Fig. 5 Hydrogen production over 1 mol% LiCl doped catalyst

Figure 6 shows the ratios of C_2H_4/C_2H_6 and CO/CO_2 against the 1 mol% LiCl doped catalysts. The presence of LiCl appears to have a conspicuous influence to the product ratios, especially doped MgO catalyst. The ratio of C_2H_4/C_2H_6 increased from 1.14 for MgO catalyst to 1.38 for 1 mol% LiCl/MgO whereas the CO/CO_2 increased from 0.14 to 0.25. For the other catalysts, no significant changes were observed in C_2H_4/C_2H_6 ratio but the CO/CO_2 ratio for CaO and SnO_2 reduced markedly due to total oxidation to CO_2 .

Because of its high selectivity to C_{2+} hydrocarbons, both the La_2O_3 and MgO have been doped with 2 mol% LiCl. Table 1 shows the results of these catalysts together with pure and 1 mol% LiCl doped catalysts for comparison purpose.

Fig. 6 Products ratio over 1 mol% LiCl doped catalyst

From Table 1, increasing LiCl concentration on MgO resulted in a negative effect where the C_{2+} selectivity decreased from 39.6% for 1 mol% LiCl/MgO catalyst to 34.7% for 2 mol% LiCl/MgO. The conversion also decreased from 27.6% to 24.7%. The most marked change was observed on the C_2H_4/C_2H_6 ratio, which decreased from 1.38 to 0.29. This was observed when Li_2CO_3 was used as a precursor. The C_{2+} selectivity increased to 42.6% with methane conversion of 29.7%, resulting in C_{2+} yield of 12.7%. The C_2H_4/C_2H_6 ratio also increased to 1.5. All the other measured parameters were kept constant.

For La_2O_3 , doping it with 2 mol% LiCl caused an increase in C_{2+} selectivity and yield which were the highest among all the catalysts studied.

Table 1: Catalytic activity of La_2O_3 and MgO based catalysts

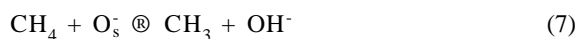
Catalysts	%CH ₄ conversion	%C ₂₊ hydrocarbons	%C ₂₊ yield	%H ₂ selectivity	%H ₂ yield	C ₂ H ₄ /C ₂ H ₆	CO/CO ₂
MgO	28.8	39.4	11.3	19.1	5.5	1.14	0.14
1 mol% LiCl/MgO	27.6	39.6	10.9	16.3	4.5	1.38	0.25
2 mol% LiCl/MgO	24.7	34.7	8.6	20.6	5.1	0.29	0.32
2 mol% Li/MgO *	29.7	42.6	12.7	17.0	5.1	1.5	0.25
La_2O_3	27.7	44.8	12.4	15.8	4.4	0.95	0.11
1 mol% LiCl/ La_2O_3	28.5	44.3	12.6	14.0	4.0	0.99	0.12
2 mol% LiCl/ La_2O_3	29.0	46.7	13.5	15.5	4.5	1.05	0.10
2 mol% Li/ La_2O_3 *	27.8	42.8	11.9	12.9	3.4	0.81	0.05

*Prepared by using Li_2CO_3 as a precursor for Li dopant

The C₂₊ selectivity and yield of 2 mol% LiCl/La₂O₃ were 46.7% and 13.5%, respectively. The H₂ selectivity and yield together with the C₂H₄/C₂H₆ and CO/CO₂ ratios did not change significantly. The activity of 2 mol% LiCl/La₂O₃ also was better than 2 mol% Li/La₂O₃ catalyst prepared by using Li₂CO₃ as the Li dopant precursor, where the C₂₊ selectivity obtained was 42.8% with methane conversion of 27.8%. The conversion, yield and selectivity values were reproducible within experimental error of ±5%.

4. DISCUSSIONS

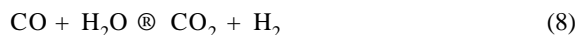
It is generally accepted that the oxidative coupling of methane to C₂ hydrocarbons and subsequently to C₃ and higher hydrocarbons is initiated by the generation of gas-phase methyl radicals^[10]. This is accomplished through the abstraction of hydrogen atom from methane, which has been proposed as follows:



where the surface oxygen species, O_s is the active site. The OH[•] would be converted to water through subsequent reactions that regenerate the O_s with the help of gas-phase oxygen. Ethane is formed via the coupling of methyl radicals in the gas phase, whereas ethylene is believed to be originated from the thermal dehydrogenation of ethane or the surface-catalyzed oxidative dehydrogenation of ethane.

Both CO and CO₂ come from the gas-phase or surface catalyzed oxidation of methane, hydrocarbon intermediate species and hydrocarbons final product. The latter two may contribute more significantly than the former for this non-selective reactions^[11]. The production of hydrogen may be invoked by these possible paths of consecutive reactions^[12]:

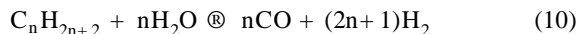
Water gas-shift reaction (referred to as WGS)



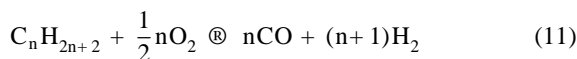
Thermal cracking of ethane



Steam reforming of hydrocarbons



Partial oxidation of hydrocarbons



Ethane dehydrogenation in the presence of steam is an un-catalyzed commercial process, whereas WGS and steam reforming of hydrocarbons do not proceed without a catalyst. Partial oxidation can occur both thermally and catalytically.

From the figures and table presented, it is noticeable that when the C₂₊ selectivity increases, the C₂H₄/C₂H₆ would also increase but not the H₂ selectivity. This indicates that the catalytic oxidative dehydrogenation of ethane and the thermal dehydrogenation of ethane are the sources of ethylene and the water gas-shift reaction (Eq. 8) is the main source of hydrogen production. The

catalytic influence on these reactions is clearly shown by the differences in activity of CaO and 1 mol% LiCl/CaO. The relative importance of the catalytic oxidative dehydrogenation of ethane and the thermal dehydrogenation of ethane is, however, difficult to determine.

The low CO/CO₂ ratio recorded on all the catalysts indicates that the steam reforming of hydrocarbons (Eq. 10) and the partial oxidation of hydrocarbons (Eq. 11) do not proceed to a significant extent. The absence of steam reforming reaction is in line with kinetics observation by Stansch^[11] on La₂O₃/CaO catalyst which stated that the reaction was not observed for reaction temperatures below 800°C but become significant above 800°C. This is because the reaction of C₂₊ hydrocarbons with water is much slower as compared with oxygen. Investigation by Hargreaves, et. al.^[12] showed that partial oxidation of hydrocarbons was found to be the dominant route to H₂ only at low oxygen conversion. In the present study, a 100% of oxygen conversion was achieved on all the catalysts system.

For the catalysts studied, the positive influence of LiCl on La₂O₃ is much more pronounced. The increase in the C₂H₄/C₂H₆ ratio may indicate the participation of chlorine in dehydrogenating ethane in the gas phase. As the calcination and reaction temperature used in this study are relatively high, significant loss of chlorine from the catalyst may occur before and definitely after the reaction. This is believed to happen because of the evaporative nature of chlorine. During experimentation, chlorine may react with water vapors resulting in the formation of HCl.

If chlorine induced dehydrogenation of ethane occurred, it is projected that MgO when doped with 2 mol% of LiCl should give a much higher C₂H₄/C₂H₆ ratio as compared to 1 mol% of LiCl/MgO. However, this is not the case in our present study. This suggests that other factor, which was influenced by the presence of Cl plays a part in the catalyst selectivity. The negative effect of chlorine on MgO is proved further when Li₂CO₃ was used as a precursor to prepare 2 mol% Li/MgO, and the C₂₊ selectivity increased together with the C₂H₄/C₂H₆ ratio. The presence of chlorine, however, possibly enhanced the C₂H₄/C₂H₆ ratio only at a much lower temperature. A study on LiCl/MgO catalyst showed the catalysts to be effective in increasing the ratio of C₂H₄/C₂H₆ up to 5 with C₂ yield of 20% at 640°C^[7].

For La₂O₃, higher doping concentration of LiCl gives a positive effect to the C₂₊ selectivity but the C₂H₄/C₂H₆ ratio remains to be similar. Again, the presence of chlorine is the main factor in the increase of C₂₊ selectivity as doping the La₂O₃ with the same Li concentration using Li₂CO₃ as the precursor do not give a similar result but a decrease in C₂₊ selectivity as observed. The different behavior of La₂O₃ and MgO based catalysts may be attributed to the influence of chlorine on the active sites of the catalysts. The chlorine seems to affect the catalysts activity more than that of lithium.

5. CONCLUSIONS

Results obtained show that the presence of LiCl has a marked influence on the activity of all the catalysts studied. The function of the chlorine atom is related more to the active sites on the surface of the catalysts rather than involved in facilitating ethylene formation via gas-phase dehydrogenation reaction of ethane, as previously suggested. It is also discovered that hydrogen is mainly produced through the water gas- shift reaction.

ACKNOWLEDGEMENTS

This research has been supported by Universiti Sains Malaysia under long-term IRPA grant (Project No. 02-02-05-7003). Megat Harun Al Rashid bin Megat Ahmad acknowledges Ministry of Science, Technology and Environment, Malaysia for award of postgraduate scholarship.

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BIOGRAPHIES

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