

Regeneration of Deactivated Ni-Catalysts for CO₂ Dry Reforming

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Abstract

The objective of the study was regeneration of Ni based catalyst that was used for DRM reaction with different mixtures of feed gas, reaction cycle time, and temperature conditions. To investigate the performance of regeneration and identify the characteristics of coke, several types of experiments has been done. Thermogravimetry (TG) and differential scanning calorimetric (DSC) analyses were used for combustion with air (20 ml/min) for temperature from 30°C to 800°C, combustion with air (20 ml/min) at different temperatures (700°C, 600°C, 550°C, and 500°C), pyrolysis with nitrogen (20 ml/min) for temperature from 30°C to 800°C, and gasification of coke with carbon dioxide (80 ml/min) for temperature from 40°C to 800°C. Simultaneous Thermal Analyzer 6000 (STA 6000, Perkin Elmer, Inc) and SDT Thermal Instrument 2960 were used for experimentation. In both cases alumina crucible pan was used which has melting point of 2000°C. It has been found that lower temperature of DRM reaction promotes deposition of coke and increases with reaction cycle time. For all the cases coke regeneration starts at around 500°C and formation of plateau has been noticed over

700°C. The coke removal performance is found almost similar at 600°C and 700°C but higher temperature intensifies the process that requires less time in comparison. Deposition of hydrogen containing volatile species was confirmed by pyrolysis reaction with nitrogen. Coke gasification using carbon dioxide showed an excellent performance which can be an added advantage of DRM technology for utilization of more greenhouse gases. Mass fraction and kinetic parameters of pseudo components such as pre-exponential factor, activation energy was determined from the developed kinetic model.

Introduction

Among the greenhouse gases (GHG), CO₂ is the major contributor that constitutes almost around 76 percent including fossil fuel, industrial processes, forestry and other land use. Next is CH₄ that accounts 16 percent. The economic and population growth are considered to be the primary driving force (IPCC, 2014). In response to this the European Commission comprising of EU-28 countries has set their goals to curb the emission. By 2020, the EU-28 countries want to reduce the GHG emission 20% comparing to the

emission of 1990 ("2020 climate and energy package | Climate Action," n.d.). By 2030 they fixed that target for 40% ("2030 climate and energy framework | Climate Action," n.d.) and by 2050 they want to reach the emission reduction level of 80% (from 1990 level) ("2050 low-carbon economy | Climate Action," n.d.). From the report of 2015, it represents that EU-28 countries already have surpassed 2020 goal with GHG emission went down to 22% of the level of 1990 ("Greenhouse gas emission statistics - emission inventories - Statistics Explained," n.d.). Now EU-28 countries must push forward to meet the goal of 2030.

Though carbon capture and storage (CCS) is a proven technology, some uncertainties are involved (Aminu, Nabavi, Rochelle, & Manovic, 2017). Another important factor to be considered is public acceptance of the technology in terms of risk perception and benefit perception (Karimi & Toikka, 2018). However, the success of CCS technology depends on public policy, government subsidy and interest that is different across the world (Thronicker, Lange, & Pless, 2016). Carbon capture and utilization (CCU) has become an attractive option. CCU refers to the capture of carbon dioxide from the sources, followed by distribution to different utilization options. It is estimated that CCU has potential of 3.7 Gigatons per year (Gt/y) which is 10% of current world emission (Koytsoumpa, Bergins, & Kakaras, 2018). This technology offers the replacement of carbon dioxide extraction from natural sources. Currently CCS technology requires subsidy and economic incentives due to having high cost. Therefore, instead of considering carbon capture as negative economic option, it also can be

preferred as an added economic value which offers reduction in cost of carbon dioxide capture (Tapia, Lee, Ooi, Foo, & Tan, 2018). Utilization of carbon dioxide can be found in various sectors (Koytsoumpa et al., 2018) which can be categorized into two main classes, i.e. for chemical feedstock, and for injection in geo-structure as fluid. Dry reforming of methane (DRM) is a promising potential technology for producing synthetic gas which offers several advantages. DRM got the attraction because of having environmental and industrial benefit (Alenazey, 2014). This technology has become lucrative due to having large boom in shale gas development resulted in the availability of large reserve of methane comparing to petroleum. DRM produces syngas (H_2 and CO), which are raw materials jointly or separately for different chemical processes for the purpose of producing energy and chemicals (Löfberg, Guerrero-Caballero, Kane, Rubbens, & Jalowiecki-Duhamel, 2017). Also, methane and carbon dioxide produced from different processes as by product can provide an added value to the existing process. DMR technology utilizes both carbon dioxide and methane directly and simultaneously which does not require the separation step. According to thermodynamic calculation CO_2 and CH_4 conversions were about 50% and 60% respectively at $300^\circ C$. So, activating both CO_2 and CH_4 at low temperature was feasible thermodynamically. Therefore, it is essential to search for the development of low temperature catalyst for DRM reaction (Wang, Yao, Wang, Mao, & Hu, 2018). Researches has found various highly active catalysts which are based on cobalt and nickel and promoted with

noble metals. However, from the economic point of view development of non-precious catalyst like Ni are more favored (Alenazey, 2014). But the DRM technology suffers from several drawbacks. High reaction temperature is required for this endothermic reaction but thermodynamically the performances are limited in most cases. Such high temperature causes sintering of active species and promotes formation of coke through methane cracking or by disproportion of CO (Boudouard reaction). And the presence of CO₂ and H₂ simultaneously causes reduction in selectivity due to unwanted water gas shift reaction (Löfberg, Kane, Guerrero-Caballero, & Jalowiecki-Duhamel, 2017).

The purpose of the study is to analyze regeneration characteristics of a Ni based catalyst that was used for DRM reaction. Thermal analysis of the catalyst regeneration will be done by changing condition and atmospheric composition. Oxygen will be used to oxidize coke in regeneration process. In addition, investigation also will be done on the performance of the reaction at different temperatures condition. To investigate the presence of hydrocarbon together with deposited coke pyrolysis reaction with nitrogen will be done. Gasification with carbon dioxide also will be explored to check the possibility of coke gasification with carbon dioxide. If this come out to be satisfactory, this will be an added value for the DRM technology. In addition, using the experimental data kinetic model will be built to identify apparent kinetic parameter of the reaction.

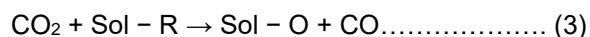
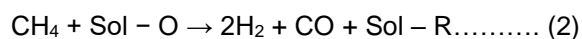
Literature Review

DRM reaction: Dry reforming of methane (DRM) is a chemical reaction that converts carbon dioxide and methane to syngas (DRM, Eq. (1)). DRM is an endothermic reaction which has optimum range of temperature between 600°C to 1000°C to have desirable level of conversion (Aramouni, Touma, Tarboush, Zeaiter, & Ahmad, 2018).



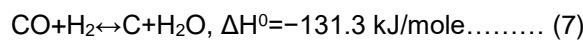
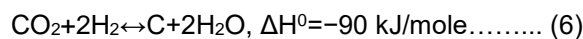
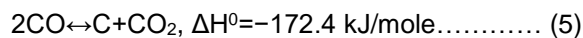
According to stoichiometry lower pressure is favorable for forward reaction. Molar ratio of CO₂ to CH₄ higher than 1, has an influence on high yield in syngas production. However, DRM is not yet mature for industrial application (Aramouni et al., 2018).

The overall DRM reaction is conducted in two stages in practical.



Here Sol-O is the oxidized catalyst and Sol-R is the reduced catalyst. In dry methane reforming reaction CH₄ and CO₂ are exposed to a solid catalyst in cyclic way. The solid catalyst acts as oxygen carrier, producing syngas while exposure to CH₄ and getting re-oxidized while exposure to CO₂. (Löfberg, Guerrero-Caballero, et al., 2017). In parallel to the reaction amid CO₂ and CH₄, several other reactions can also occur such as decomposition of methane (Eq. (4)), disproportion of carbon monoxide which is also known as Boudouard reaction (Eq. (5)), hydrogenation of carbon dioxide (Eq. (6)), and hydrogenation of carbon monoxide (Eq. (7)).

These reactions are responsible to form carbon during DRM. The reverse water gas shift (RWGS) reaction (Eq. (8)) is dependent on specific temperature range equilibrium which exist during DRM reaction. Over 800°C, Boudouard and RWGS reaction will occur (Aramouni et al., 2018).



The Gibbs free energy calculation dictates that CH₄ decomposition mainly occurs higher than 550°C temperature and Boudouard reaction occurs mainly below 700°C temperature. Therefore, the temperature range 550°C to 700°C is responsible for coke formation. The deposition of coke also influenced by carbon, hydrogen, and oxygen ratio in the raw gas. Lower ratios of O/C and H/C promotes the tendency of higher coke formation. Therefore, dry reforming is more prone to coke deposition than steam reforming (Gao, Jiang, Meng, Yan, & Aihemaiti, 2018).

Catalyst deactivation and regeneration:

Catalyst deactivation is a complex process. It depends on several factors such as reactant and product of the reaction, catalytic material, reaction temperature and pressure, reaction mechanism. Fouling, poisoning, and sintering are some of the processes of catalyst deactivation. Fouling occurs when reactants, or intermediates, or products in the reactor are deposited on the catalyst surface, resulting in blocking of catalyst active sites. Fouling by carbonaceous species

also known as "coking" is the most common type. Coke deposition can occur in several forms, including metal carbides, polymer aggregates, tar, and laminar graphite. Poisoning is a result of chemisorption by compounds of the process stream which modify or block the active sites on the catalyst. The poison may bring changes in morphology of catalyst surface in several ways such as reconstructing surface or relaxation of surface, modifying bond between support and metal catalyst. Poison toxicity is dependent on the adsorption enthalpy of the poison, and free energy of the adsorption process, which regulates the equilibrium constant of poison chemisorption. Sintering is a result of agglomeration and growth of small crystals which forms in the support or catalyst. The rearrangement of catalyst structure due to sintering causes a decrease in catalyst surface area, as a result loss of active sites. Sintering usually occurs when local temperature is approximately above one-third to one-half of catalyst melting temperature (T_m). For "dry" condition, upper limit of temperature ((1/2) T_m) is applied and when steam is present lower limit of temperature ((1/3) T_m) is applied, as steam causes reorganization of metals such as alumina, silica. Prevention of sintering can be done by doping catalyst with stabilizers so that to have high melting points to prevent small crystal agglomeration. The high melting point materials such as magnesia, alumina, and chromia are usually doped as stabilizers. Sometimes it is likely to restore the activity of catalyst completely or partially by chemical treatment. The process of regeneration can be slow, either because of thermodynamic limitation or resistance in

diffusion due to blockage of pores in catalyst. Though at high temperature the desorption rate usually increases, prolonged exposure to high temperature of catalyst can result in sintering and loss of activity which is irreversible. If it is not possible to gasify the deposited species at lower temperature (less than sintering temperature), then the fouling or poisoning is likely to be irreversible (Missen, Mims, & Saville, n.d.).

Experimental Setup

In this work regeneration of Ni-based catalyst was analyzed that was used in dry reforming of methane. The preparation method of the catalyst is described in (Dębek et al., 2016). The whole experimental activity can be categorized in four major group: (a) combustion with air (20 ml/min) up to 800°C, where the temperature was raised from 30°C to 800°C at a rate of 10°C per minute and kept in isothermal condition for 10 minutes, (b) combustion with air (20 ml/min) at different temperatures (700°C, 600°C, 550°C, and 500°C), where the temperature was raised from 30°C to preferred temperature at a rate of 10°C per minute and kept in isothermal condition for 60 minutes, (c) pyrolysis with nitrogen (20 ml/min), where the temperature was raised from 30°C to 800°C at a rate of 10°C per minute and kept in isothermal condition for 60 minutes, and (d) gasification with carbon dioxide, where the temperature was raised from 40°C to 800°C temperature at a rate of 10°C per minute and kept in isothermal condition for 60 minutes. Simultaneous Thermal Analyzer 6000 (STA 6000, Perkin Elmer, Inc) was used for the first three categories of experiment and SDT Thermal Instrument 2960 was used for gasification with carbon dioxide. In both cases

alumina crucible pan was used which has melting point of 2000°C. The results of the experiments are analyzed in three broad categories such as: (a) thermal decomposition analysis, (b) energy profile analysis, and (c) kinetic model development to identify apparent kinetic parameter.

Result and Discussion

Thermal decomposition analysis

Combustion with air: From the graph, Figure 1 it can be observed that for all sample's decomposition starts at around 500°C. The weight of the sample remained stable after 700°C. In general, for all samples, large amount of coke deposition has been witnessed but the amount depends on the temperature of the reaction environment as well as duration of the reaction. For the case of the samples "750°C-24h-spent" and "550°C-24h-spent", the mass reduction was around 72% and 91% respectively and that for 750°C-5h-spent and 550°C-5h-spent were around 55% and 80% respectively.

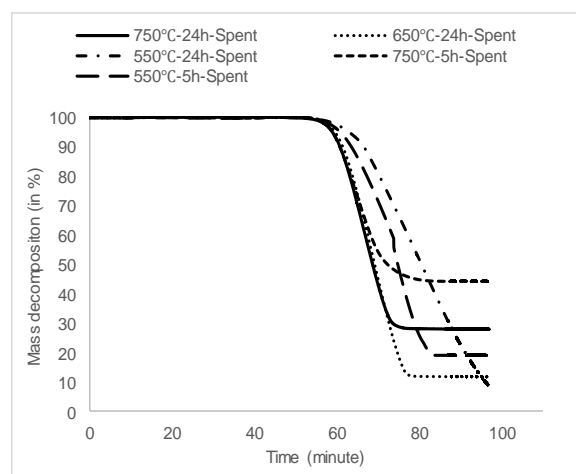


Figure 1: TG analysis of all samples up to 800°C.

Pyrolysis with nitrogen: In the DRM reaction methane molecules goes through several steps

of cracking processes for dissociation of hydrogen from methane molecules. Therefore, there is possibility of accumulation of hydrogen containing volatile species along with coke during DRM reaction as methane decomposition reaction is influential in the overall process and conversion of methane is lower than the conversion of carbon dioxide (Dębek et al., 2015). All the samples were investigated through pyrolysis reaction to identify the presence of hydrogen bearing volatile species together with deposited coke in the oxygen free environment.

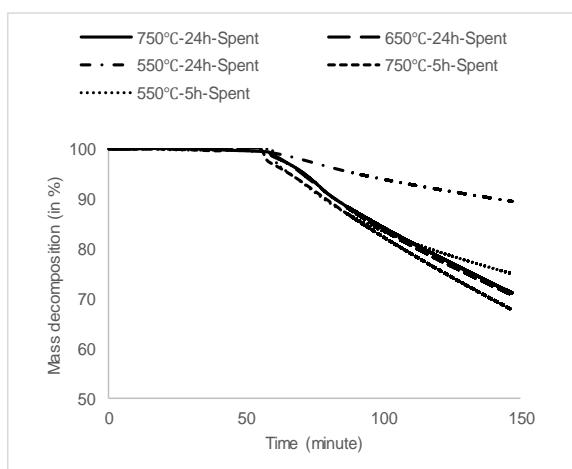


Figure 2: TG analyses of pyrolysis reaction with nitrogen of all samples.

Figure 2 represents thermogravimetry graph which depicts that the reaction or mass decomposition starts at around 500°C. The noticeable fact is that the rate of mass degradation was almost linear with function of time. As described above in the temperature programming that the sample was heated up to 800°C, we did not analyze thermal decomposition at higher temperature. Although the sample was heated up to 800°C, the tendency of the degradation curve signifies that the sample could degrade much with higher temperature. So, the result of the pyrolysis reaction implies the

possibility of deposition of hydrogen containing volatile together with coke during the time of DRM reaction.

Coke gasification with carbon dioxide: From sustainability and environmental point of view, if DRM technology can be developed industrially, it will have a potential impact in reduction of greenhouse gas emission. On the other side the main drawback of this technology for not yet being industrially developed is that in dry reforming of methane reaction, coke deposition is inevitable. Therefore, it will be an added advantage of the technology, if deposited coke can be gasified with carbon dioxide. It will ensure further utilization of carbon dioxide. So the deposited coke can be gasified on line by using a gasifying agent CO₂ for the better performance of catalytic reactor (Alenazey, 2014).

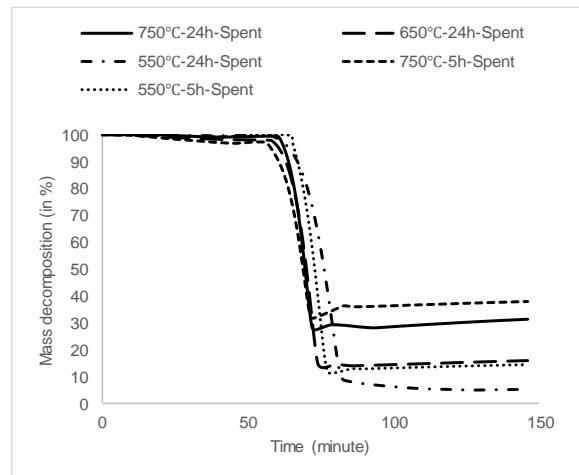


Figure 3: TG analysis of all samples for gasification of coke with carbon dioxide.

The Figure 3 represents the thermogravimetry of regeneration of catalyst through gasification reaction with carbon dioxide. From the figure it can be noticed that the decomposition starts at around 550°C and the slope represents that after starting the decomposition it continued abruptly.

Another significant observation is that above 700°C, there is a slight increase in mass. It might be a reaction that transform the gas phase into solid phase over 700°C. Coke gasification reaction with carbon dioxide also infers that at lower temperature of DRM reaction coke deposition tendency is much higher. The most important finding is that the possibility of regeneration with carbon dioxide. Catalyst regeneration with carbon dioxide showed an excellent performance with 95% mass decomposed for the sample “550°C-24h-spent”. For the samples “750°C-24h-spent” and “750°C-5h-spent” the percentage of mass decomposition was around 68 and 62 respectively.

Energy profile analysis: The following figures in this section describes heat absorbed or released during the heating process and the heat flow data is provided by differential scanning calorimetry (DSC) signal. Using heat flow data, the physical and chemical activity can be anticipated but not in a certain manner.

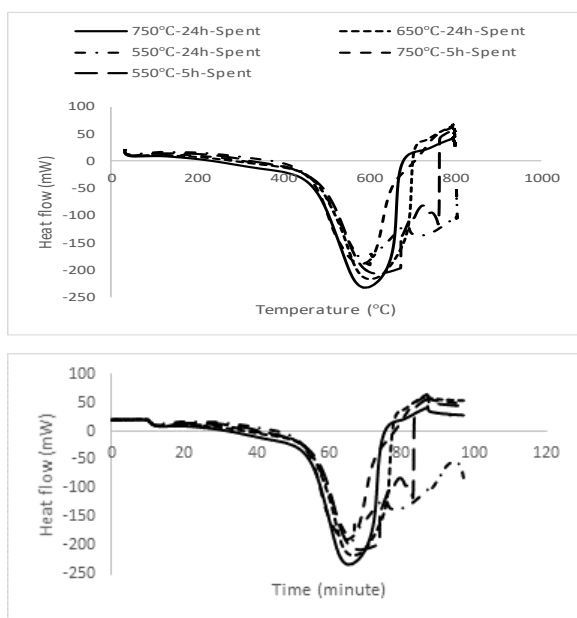


Figure 4: Heat flow during combustion reaction with air (30°C to 800°C).

The Figure 4 above shows heat flow in terms of temperature and time for combustion reaction of all sample up to 800°C. It is noticeable in the figure that at around 400°C the sample shows starting of an exothermic trend with a slow process. At around 500°C, partial oxidation of coke can also be noticed which continues with a higher reaction rate with the rise in temperature. The peak of the reaction reaches at around 600°C, after that it started to decrease. From the thermogravimetry analysis curve (Figure 1) it also noticed that the fractional mass degradation starts at around 500°C. The degradation continued to increase almost in a smooth rate linearly.

So, it can be generalized that at around 400°C, decomposition of large molecule starts but it does not vaporize. That is why no change can be seen in the thermogravimetry curve (Figure 1) at around 400°C temperatures. With the rise in temperature the fragmented small molecules start to vaporize at around 500°C and it continues with the increase in temperature and becomes faster. After the exothermic reaction the baseline went little up comparing to pre-exothermic reaction baseline. This indicates that the post-combustion components have much higher heat capacity.

From heat flow curve it also can be interpreted that coke deposition occurs in a layered manner. Therefore, overlap in the reaction peak is noticeable. Another distinguishable fact is that the catalysts used in higher temperature environment around 650°C or above, less variety of coke species are deposited. In terms of characteristics, these components are not much different. For low

temperature reaction environment (550°C) a much larger variety of coke species are deposited.

Kinetic model fitting and parameter estimation:

The idea behind the kinetic model is to compute kinetic parameters of reaction kinetics. In kinetic model simulation, it was assumed that pseudo components are thermally degraded through various reaction such as: combustion with air, pyrolysis with nitrogen, and gasification with carbon dioxide. In addition, it was also assumed that the conversion of pseudo components follows first order reaction and independent of each other. The pseudo components were calculated using Euler’s method. Least square method was used to determine pre-exponential factor, activation energy, and mass fraction of each pseudo component. Generalized Reduced Gradient (GRG) nonlinear algorithm was used in solver tool of Microsoft Excel.

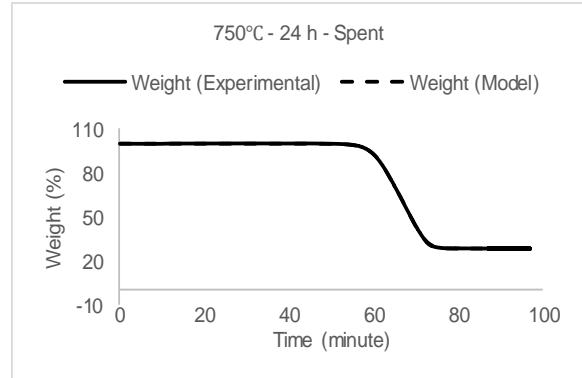


Figure 5: Model fitting for combustion reaction with air up to 800°C of sample “750°C-24h-Spent”.

The Figure 5 exhibits a good fit between the curves based on experimental data and model data of sample “750°C-24h-Spent”. From Table 1, the kinetic parameter and pseudo component fractions are provided. It can be noticed that in in Table 1 that in all reactions two types of pseudo components are found except for the sample “550°C-5h-Spent” where only one pseudo component is deposited. Though two pseudo components have been found for the sample “750°C-5h-Spent”, the kinetic parameters are very close.

Table 1: Kinetic parameters of combustion reaction with air up to 800°C of all samples

Sample name	750°C-24h-Spent	650°C-24h-Spent	550°C-24h-Spent	750°C-5h-Spent	550°C-5h-Spent
Component 1 (%)	11.52	40.19	47.345	33.63	80.913
K (1/min)	1.36E-10	1.90E-08	3.50E-07	3.18E-08	4.80E-07
Ea (kJ/mole)	54508.17	39024.2	27604.41	38239.85	26514.73
Component 2 (%)	60.42	47.963	44.05	22.06	0
K (1/min)	1.94E-08	2.59E-12	6.87E-16	1.46E-07	0
Ea (kJ/mole)	36770.314	54362	61549.65	30861.62	0

Conclusion

It has been analyzed that lower temperature of DRM reaction promotes deposition of coke and intensifies with elongation of time. In general, decomposition starts above 500°C at around 700°C all samples showed a plateau. The amount of coke regeneration at around 600°C and 700°C

is almost same but the only difference is at higher temperature the reaction occurs faster. The pyrolysis reaction with nitrogen indicates the possible deposition of hydrocarbon species together with deposited coke. More interestingly coke gasification with carbon dioxide shows and excellent performance to regenerate the catalyst. This is an added advantage of DRM to reduce these greenhouse gases. From the heat flow analysis, it can be noticed by the overlap of the peak that coke is deposited in stratified structure. Therefore after removal one layer another layer starts to decompose. The kinetic model fitting shows that two types of pseudo components are deposited as coke. At higher temperature of regeneration, both pseudo components can be removed while at lower temperature only one pseudo component can be removed.

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