

**SYNTHESIS AND CHARACTERIZATION OF EFB CLINKER SUPPORTED  
NICKEL CATALYST FOR SYNGAS PRODUCTION FROM REACTIVE  
FLUID MIXTURE OF CO<sub>2</sub>-CH<sub>4</sub>**

**(PENGHASILAN DAN PENCIRIAN KATALIS KLINKER EFB-NIKEL UNTUK  
PENGHASILAN GAS SINTETIK DARIPADA CAMPURAN BENDALIR  
REAKTIF CO<sub>2</sub>-CH<sub>4</sub>)**

**CHENG CHIN KUI  
MD. MAKSUDUR RAHMAN KHAN  
CHIN SIM YEE  
SUMAIYA BT ZAINAL ABIDIN  
JOLIUS GIMBUN**

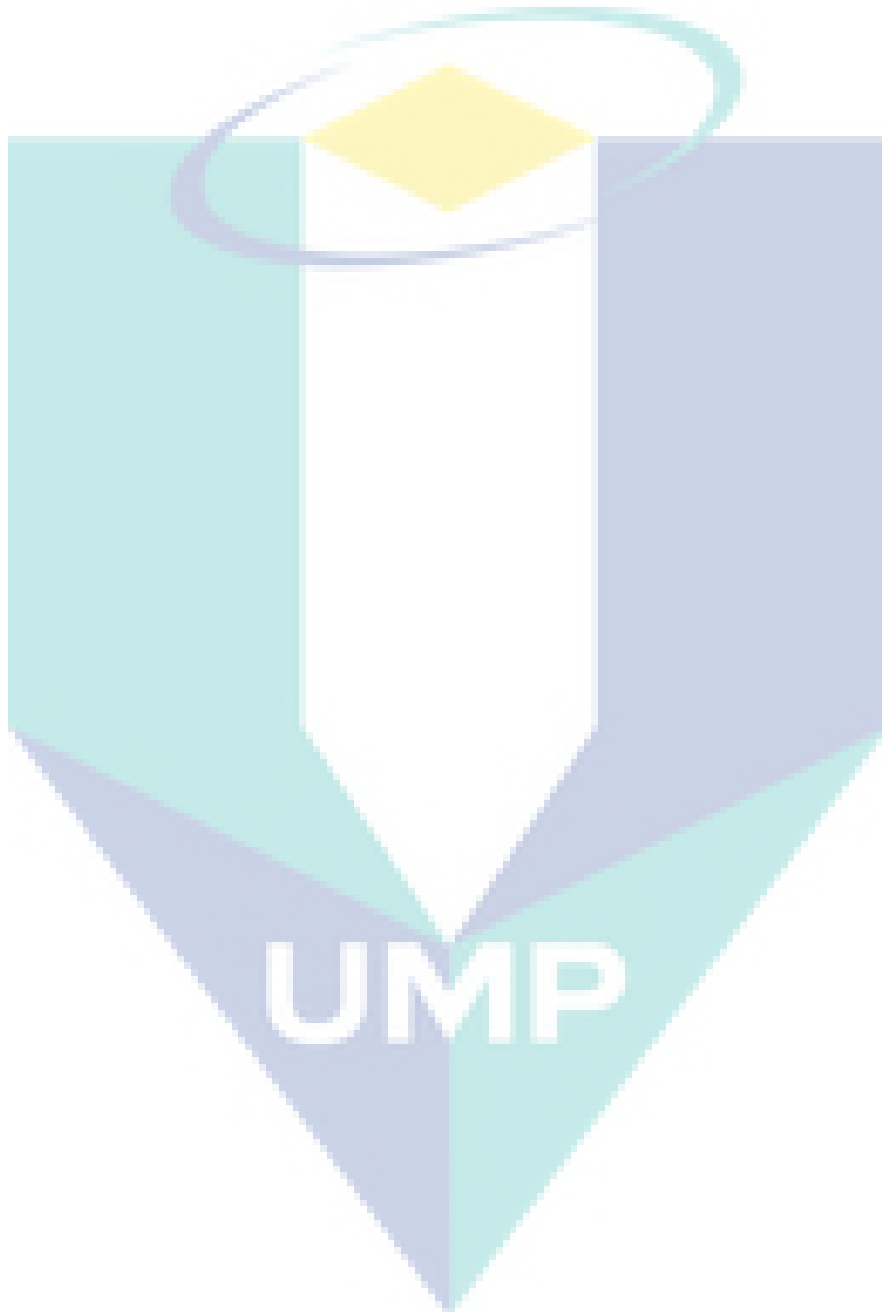
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# SYNTHESIS AND CHARACTERIZATION OF EFB CLINKER SUPPORTED NICKEL CATALYST FOR SYNGAS PRODUCTION FROM REACTIVE FLUID MIXTURE OF CO<sub>2</sub>-CH<sub>4</sub>

*(Key words: Dry reforming; Empty fruit bunch (EFB) clinker; Synthesis gas)*

Methane dry reforming is environmental friendly due to the potential sequestration of CO<sub>2</sub>. Therefore, substantial research works have been carried out to develop reforming catalysts that exhibit high catalytic performance and excellent longevity. The current work reports on the synthesis of 20wt%Co/80wt%EFB clinker and 20wt%Ni/80wt%EFB clinker catalysts via wet-impregnation technique, followed by various characterization techniques such as BET, XRD, XRF, TGA and FESEM. The BET specific surface area of Co/EFB clinker and Ni/EFB clinker catalyst were 2.83 m<sup>2</sup>g<sup>-1</sup> and 2.37 m<sup>2</sup>g<sup>-1</sup>, respectively. In addition, XRD diffractogram of EFB clinker showed two highest peaks at around  $2\theta = 27.2^\circ$  and  $31.5^\circ$ , which can be attributed to CaO and K<sub>2</sub>O species, respectively. This finding was also consistent with XRF analysis where potassium (K) and calcium (Ca) were positively identified. Subsequently, methane dry reforming studies revealed that Ni/EFB clinker catalyst always yielded higher formation rates of H<sub>2</sub> and CO. Indeed, the rate was maximum at CO<sub>2</sub>:CH<sub>4</sub> feed ratio of unity. The average conversion of CH<sub>4</sub> for reactions using Co/EFB clinker and Ni/EFB clinker catalyst are obtained to be 78.26 % and 78.19 % respectively. Meanwhile, the average conversion of CO<sub>2</sub> for reactions using Co/EFB clinker and Ni/EFB clinker catalyst are 80.40 % and 81.17 % respectively. When reaction temperatures were varied, the activation energy over Co/EFB clinker was 124.4 kJ mol<sup>-1</sup> while that for the reaction over the Ni/EFB clinker catalyst was 113.0 kJ mol<sup>-1</sup>. Post reforming reaction, oxidation profiles of the used catalysts indicated carbon-free condition. This was corroborated by the FESEM images for both sets of used catalysts, where no carbon whisker was found.

**Key researchers :**

**Cheng Chin Kui**  
**Md. Maksudur Rahman Khan**  
**Chin Sim Yee**  
**Sumaiya Bt Zainal Abidin**  
**Jolius Gim bun**

**E-mail :** *chinkui@ump.edu.my*  
**Tel. No. :** 09 549 2896  
**Vote No. :** 09 549 2889

## ABSTRAK

Tindak balas metana dan karbon dioksida merupakan satu proses yang mesra alam. Oleh itu, banyak penyelidikan telah dijalankan bagi menghasilkan pemangkin yang berprestasi tinggi dan berhayat panjang. Kerja ini bertujuan untuk menghasilkan pemangkin yang berasaskan Ni dan Co yang disokong oleh klinker tandan buah sawit melalui kaedah impregnasi basah dengan formulasi yang 20-80. Pelbagai teknik pencirian seperti BET, XRD, XRF, TGA, FESEM dan EDX telah digunakan untuk mendapatkan ciri-ciri pemangkin yang dihasil. Kajian BET mendapati bahawa kawasan permukaan tertentu BET untuk pemangkin Co/EFB klinker dan Ni/EFB klinker adalah  $2.83 \text{ m}^2\text{g}^{-1}$  and  $2.37 \text{ m}^2\text{g}^{-1}$  masing-masing. Di samping itu, diffractogram XRD EFB klinker menunjukkan dua puncak di  $2\theta = 27.2^\circ$  dan  $31.5^\circ$ , yang boleh dikaitkan dengan spesies  $\text{CaO}$  dan  $\text{K}_2\text{O}$ . Carian ini juga konsisten dengan analisis XRF iaitu kalium (K) dan kalsium (Ca) dapat dikenal pasti secara positif. Selain itu, kajian tindak balas metana dan karbon dioksida mendedahkan bahawa pemangkin Ni/EFB klinker sentiasa menghasilkan kadar penghasilan  $\text{H}_2$  dan  $\text{CO}$  yang lebih tinggi. Sesungguhnya, kadar itu adalah maksimum pada  $\text{CO}_2:\text{CH}_4$  bernisbah 1:1. Penukaran purata  $\text{CH}_4$  untuk tindak balas menggunakan pemangkin Co/EFB klinker dan Ni/EFB klinker didapati ialah 78.26% dan 78.19% masing-masing. Sementara itu, penukaran purata  $\text{CO}_2$  untuk tindak balas menggunakan pemangkin Co/EFB klinker dan Ni/EFB klinker didapati ialah 80.40% dan 81.17% masing-masing. Apabila suhu tindak balas telah diubah, tenaga pengaktifan bagi Co/EFB klinker adalah  $124.4 \text{ kJmol}^{-1}$  manakala bagi tindak balas yang pakai pemangkin Ni/EFB klinker adalah  $113.0 \text{ kJmol}^{-1}$ . Kedua-dua pemangkin yang telah dipakai digunakan untuk menjalankan analisis profil pengoksidaan. Analisis bagi kedua-dua pemangkin mendapati bahawa kedua-dua set pemangkin yang diguna tidak menghadapi masalah pemendapan karbon yang kritikal.

# 1 INTRODUCTION

## 1.1 Background

Synthesis gas is a fuel gas mixture comprised of CO and H<sub>2</sub>. It is a vital feedstock for downstream petrochemical industries i.e. gasoline, ammonia, methanol etc. As the world grapples with potentially-devastating energy crunch situation, renewable energy such as synthesis gas has been touted as a potential saviour.

Significantly, in Malaysia palm oil mill effluent or also known as POME is a highly polluting wastewater with high chemical oxygen demand (COD) and biochemical oxygen demand (BOD) (Lam & Lee, 2011). Under ambient conditions, degradation of POME usually releases a large amount of CH<sub>4</sub> and CO<sub>2</sub> (collectively known as biogas) into the atmosphere, which may worsen the global warming (Schuchardt et al., 2008). Carbon dioxide (CO<sub>2</sub>) has been identified as one of the most significant greenhouse gases arising from anthropogenic activities. Hence, mankind needs to curb CO<sub>2</sub> emissions in order to counteract global warming (Er-rbib et al., 2012). One of the propositions is to utilize CH<sub>4</sub> and CO<sub>2</sub> simultaneously via dry (CO<sub>2</sub>) reforming pathway. The dry reforming of methane results in product with a lower H<sub>2</sub>:CO ratio (< 2.0) which is more favourable for olefins or methanol synthesis compared to the yield from the conventional steam reforming which normally produces H<sub>2</sub>:CO ratios > 3.0 (Siahvashi & Adesina, 2013). Besides, the use of CO<sub>2</sub> as co-reactant has also attracted growing attention due to the potential sequestration of CO<sub>2</sub> and energy savings (Siahvashi & Adesina, 2013).

Indeed, catalysts play a vital role in the aforementioned reaction. Catalyst is usually comprised of active metal and support. A supporter is usually a solid with a high surface area, to which a catalyst is affixed. Relentless efforts have been dedicated to maximize the surface area of a catalyst by distributing it over the support (McNaught & Wilkinson, 2014). Previous studies and reviews have been published on the Ni based metal with different supported catalysts employed in the light hydrocarbon dry reforming. An evaluative investigation of propane CO<sub>2</sub> reforming over bimetallic alumina supported 5%Mo-10%Ni catalyst has been carried out in a fixed bed reactor at 0.1 M Pa and temperatures ranging from 823 – 973K. It was found that Mo-Ni is a stable and active catalyst at different temperature during the dry reforming process (Siahvashi et al., 2013). Besides, numerous supported catalysts have been tested, especially Ni- and noble metal-based catalysts, and they have been found to exhibit promising catalytic

performance. Conversion of  $\text{CH}_4$  and  $\text{CO}_2$  to synthesis gas, approaching those defined by thermodynamic equilibrium, can be obtained over the aforementioned catalysts as long as sufficient contact times are maintained.

One of the major problems encountered during dry reforming is the deactivation of the catalysts due to carbon deposition. To alleviate the deactivation problem arising from Ni catalysts coking, it has been shown that addition of alkali or alkaline earth oxide to the Ni based catalyst lead to drastic decrease in the number of sites for carbon formation, while sufficiently maintaining the active sites for the reforming (Lemonidou et al., 1998). From the EDX result of EFB clinker, it was found that EFB has a basic characteristic and it should be suitable to act as the supporter for the Ni-based and Co-based catalysts.

## ***1.2 Problem Statement and Motivation***

Oil palm is the most important commodity from Malaysia with significant contribution to its gross domestic product (GDP). Lignocellulosic biomass which is produced from the oil palm industries include parts such as oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB) and palm oil mill effluent (POME). Nevertheless, the presence of these oil palm wastes has created a major disposal problem (Abdullah & Sulaiman, 2013). EFB are the main source of biomass that could be utilized by the power plant due to its ready-availability as daily biomass-waste. Most of the plantations use a small fraction of this EFB waste for internal use, with a large portion being put aside for either biodegrading into compost, or at times burnt to avoid space loss by storage (Matt et al., 2014). As a fuel, EFB is burnt in a furnace to generate heat and electricity for sustaining palm oil mill operation. The residue from this process is what is known as EFB clinker whereby it will be adopted for the first time in this work as supporter for Ni catalyst for methane dry reforming reaction. Most of the previous studies primarily investigated the development of catalyst for high catalytic performance and stability. Therefore, an optimum conversion of synthesis gas can be achieved. However, most of these works did not focus on the environment impact of the catalyst. To illustrate, an evaluative investigation of propane  $\text{CO}_2$  reforming over bimetallic alumina supported 5%Mo-10%Ni catalyst sounds promising in dry reforming (Siahvashi et al., 2013). In fact, calcium aluminate has been proven to be active with lower coke deposition for the reaction of reforming (Lemonidou et al., 1998). From the characterisation of EFB clinker, high percentage of K and Ca were found. Hence it is believed to be basic. A basic catalyst can reduce back pressure in the packed bed column as carbon deposition will be reduced substantially (Ranjbar & Rezaei, 2012b). Last but not least, it

is worth to explore the possibility of this palm oil industries waste as catalyst supporter in this study.

### ***1.3 Objective***

This research work is carried out to synthesize and characterize EFB clinker supported 20wt% Co and 20wt%Ni catalysts for methane dry reforming studies at atmospheric pressure.

### ***1.4 Scopes***

In order to achieve the outlined objective of this work, the following scopes have been identified:

i) **Catalysts Preparation:**

The 20wt%Co/80wt%EFB and 20wt%Ni/80wt%EFB catalysts were prepared via wet-impregnation method.

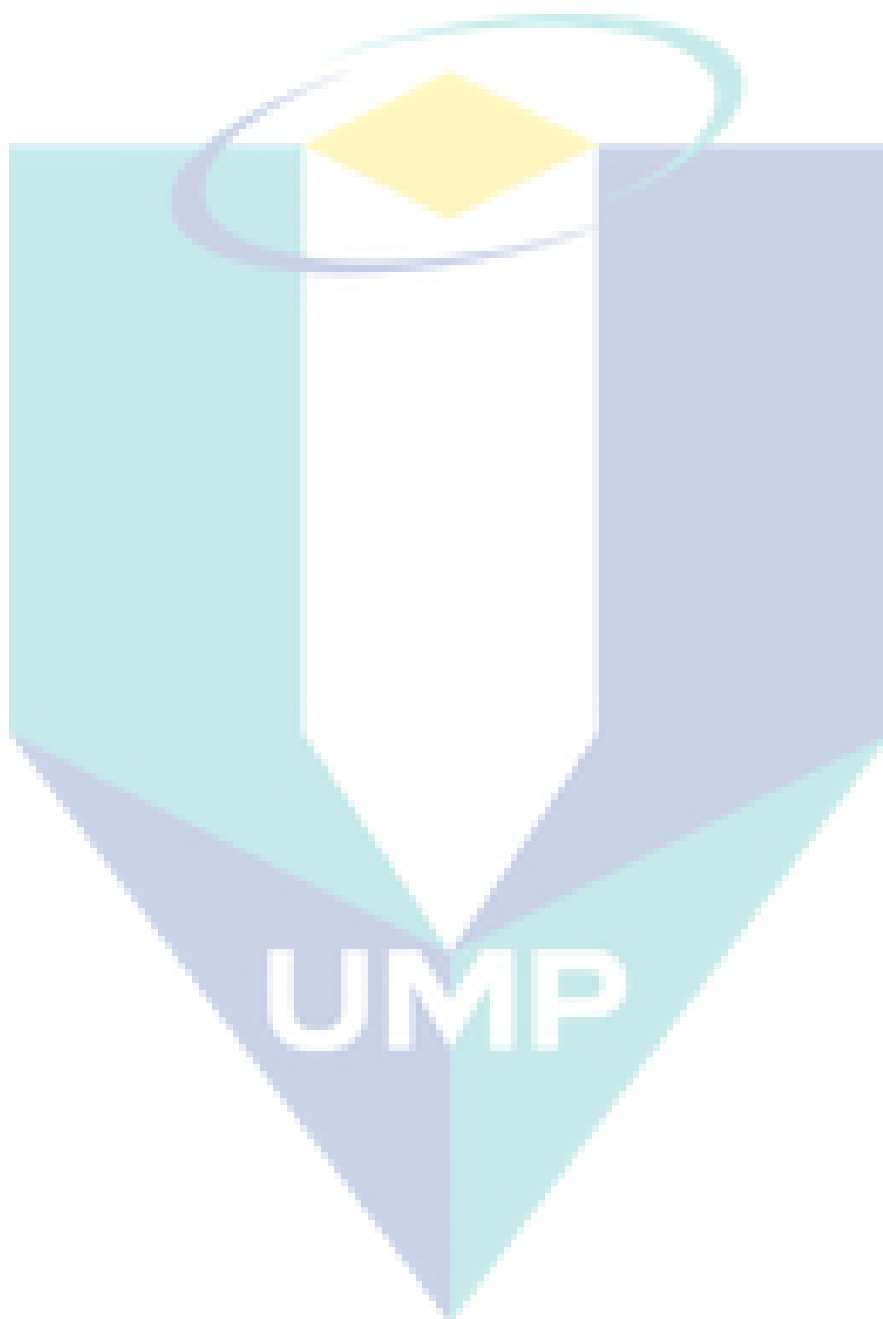
ii) **Catalyst Characterisation:**

The methods applied in this study were BET, XRD, XRF, TGA, FESEM and EDX. BET was used to find out the specific surface area, pore volume and pore size distribution sample of catalysts. The crystalline structure and the size of crystallite diameter were determined via XRD. Besides, XRF was employed for analysis of major and trace elements in the EFB clinker. The catalyst-gas interaction profile was obtained from TGA instrument. In addition, TGA also allowed the determination of coke location as well. Lastly, the image of coke deposit structure at the sample catalysts can be observed via FESEM while EDX shows the composition of element that contained in the sample catalysts.

iii) **Catalyst Reaction:**

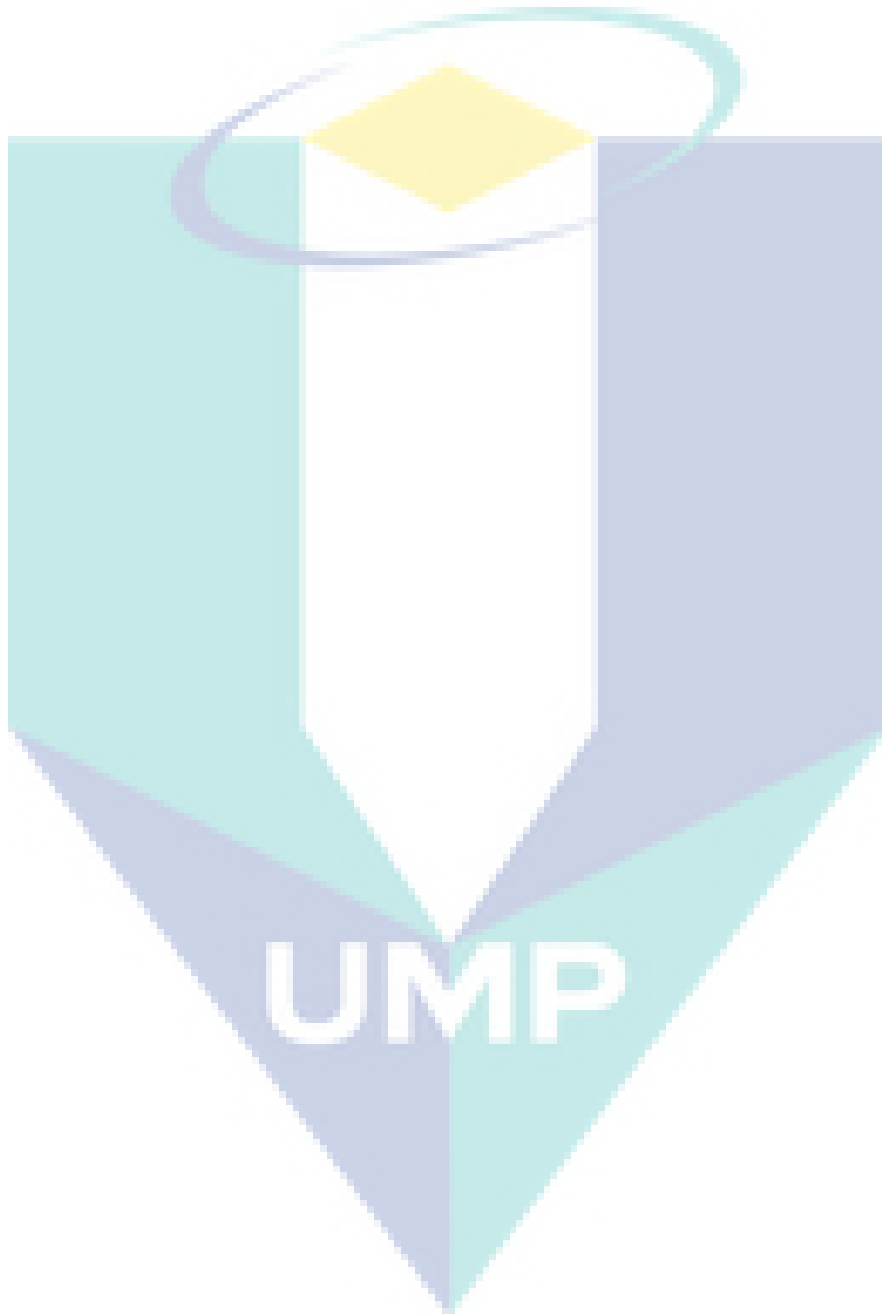
The catalytic screening was carried out by placing 0.3 g of catalyst into the stainless steel fixed-bed reactor (ID: 10 mm) supported by two layers of quartz wool. The feed comprised of the CH<sub>4</sub> and CO<sub>2</sub> mixture at equal proportion and the reaction temperature was set at 1173 K for catalyst screening studies. Subsequently, the CO<sub>2</sub> to CH<sub>4</sub> ratios

and temperatures were varied between 1073 – 1173 K to determine the ratio of product produced by the best catalyst.





**2 TECHNICAL PAPER**





# Potential of empty fruit bunch clinker as a support for nickel and cobalt catalysts in methane dry reforming: waste to wealth approach

Chin Kui Cheng\*, Han Jie Chan

Faculty of Chemical & Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Kuantan, Pahang, Malaysia

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## ABSTRACT

The current work reports on the synthesis of 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalysts via wet-impregnation technique, followed by various characterization techniques such as BET, XRD, XRF, TGA and FESEM. The BET specific surface area of 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalyst were 3.85 m<sup>2</sup>/g and 3.88 m<sup>2</sup>/g, respectively. In addition, XRD diffractogram of EFB clinker showed two highest peaks at around  $2\theta = 27.2^\circ$  and  $31.5^\circ$ , which can be attributed to CaO and K<sub>2</sub>O species, respectively. This finding was also consistent with XRF analysis where potassium (K) and calcium (Ca) were positively identified. Subsequently, methane dry reforming studies revealed that the average conversion of CH<sub>4</sub> for both sets of catalysts was nearly similar at 80.0%. When reaction temperatures were varied, the activation energy over the Co-based catalyst was 124.4 kJ/mol while that for the reaction over the Ni-based catalyst was 113.0 kJ/mol. In addition, during 12 days of dry reforming reaction, the conversion of CH<sub>4</sub> was relatively stable over both sets of catalysts indicative of stability of their properties.

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## 1. Introduction

Tropical countries such as Malaysia and Indonesia with abundant arable lands are home to large acreages of oil palm plantations [1,2]. Collectively, the processing of crude palm oil requires the utilization of steam for bunch sterilization and extraction of oil from the fresh fruit palm [3]. This steam is generated from the burning of empty fruit bunch (EFB) in a high temperature furnace, which is often accompanied by the formation of clinkers as waste [4]. Several works on the sustainable use of EFB clinker has been reported. Empty fruit bunch clinker has been reportedly used to reinforce concrete as well as used as catalyst in biodiesel production [5–7].

One of the ways to sustainably use EFB clinker is by utilizing it as catalyst support. Characterization studies by Yaakob et al. [6] showed that the EFB clinker is made up of about 62% of CaO, and 10% of K<sub>2</sub>O, which can enhance the dispersion of the active metal on the support. The use of synthesized metal catalyst on alkaline support such as MgO and CaO for dry reforming of methane has been reported [8]. The findings from this study show that MgO and CaO support reduce carbon deposition and enhanced the stability of the catalyst due to their basicity [8]. Amongst the different

methane reforming processes that have been investigated using alkaline supported catalyst, dry reforming has gain wide interest. This is due to the utilization of CO<sub>2</sub> and CH<sub>4</sub>, two main components of greenhouse gases as feedstock [9].

Dry (CO<sub>2</sub>) reforming of methane is a well-studied reaction that is of both scientific and industrial importance. This reaction produces syngas (cf. Eq. (1)) which serves as feedstock for production of wide range of products using Fischer Tropsch synthesis (FTS) [10].



Conventional production of syngas is via natural gas steam reforming [11]. Steam reforming is presently employed in commercial production of hydrogen and syngas over alkaline promoted nickel catalyst [12]. However, challenges such as catalysts deactivation from sintering, carbon deposition and poisoning has aroused the interest of researchers finding a more suitable and stable catalysts for syngas production [13]. Furthermore, steam reforming process does not have the capacity to mitigate greenhouse gases.

Several metal-based catalysts has been investigated for dry reforming of methane using convectional supports such as Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, MgO and TiO<sub>2</sub> [14–16]. These supports have been reported to provide a good thermal stability for the catalysts thereby reducing the degree of catalysts deactivation [17]. However, due to acidic nature of supports such as SiO<sub>2</sub> and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, lower activities were reported for these supports. Hence, these supports

\* Corresponding Author. Tel.: +60 9 549 2896; fax: +60 9 549 2889/+60 9 549 2896.

E-mail address: [chinkui@ump.edu.my](mailto:chinkui@ump.edu.my) (C.K. Cheng).

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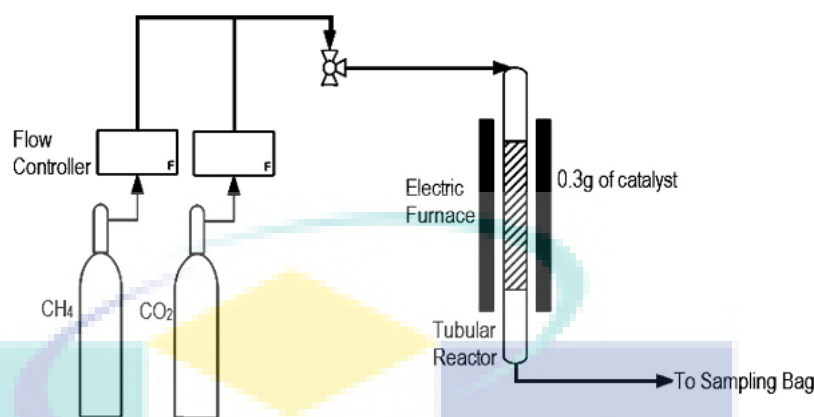


Fig. 1. Experimental rig layout for conducting methane dry reforming reaction.

with acidic properties would not be suitable for reaction involving acidic gas such as  $\text{CO}_2$ . Significantly, the use of unconventional catalysts such as EFB clinker has not been investigated for dry reforming of methane. Besides constituting about 62% of  $\text{CaO}$  which makes it a potential basic support, its utilization would help reduce health risk in the environment. In this study, the use of EFB clinker as catalyst support for the impregnation of Ni and Co metals for methane dry reforming application was assessed. Furthermore, the physicochemical properties of the support as well as its role in enhancing the thermal stability and catalytic activity were also being investigated.

## 2. Materials and methods

### 2.1. Catalysts synthesis

The empty fruit bunch (EFB) clinker, which was sourced locally, was crushed using a mortar grinder. It was then sieved to particle diameters that ranged from 100 to 250  $\mu\text{m}$ . Subsequently, 16.0 g of EFB clinker was stirred rigorously with solution containing cobalt nitrate ( $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) (equivalent to 4.0 g of Co) for 3 h to produce 20 wt% Co/80 wt% EFB clinker. Another set of catalyst, which employed nickel nitrate ( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) (equivalent to 4.0 g of Ni) as catalyst for 20 wt% Ni/80 wt% EFB clinker catalyst, was also prepared using the same procedure, in the presence of 16.0 g of EFB clinker. This formulation was typical of metal loading required for catalyst synthesized via wet-impregnation procedure and also reported before by Cheng et al. [18,19]. Post impregnation, the slurry was oven-dried at 403 K for overnight, followed by calcination at 1073 K for 6 h employing a ramping of 10 K/min.

### 2.2. Fresh catalyst characterization

The metal-EFB clinker support interaction was studied from the thermal-programmed calcination (TPC) of the fresh dried catalyst using a TGA Q-500 instrument. Non-isothermal temperature-programmed calcination was carried out from room temperature to 1173 K with heating rates at 10 K/min and a continuous flow of 50 ml/min of high purity air. The BET specific surface area and pore-size distribution of the freshly-calcined catalysts were obtained from the multipoint Brunauer-Emmett-Teller (BET) method. The raw isotherm data were sourced from  $\text{N}_2$  physisorption at 77 K conducted in a Thermo Scientific Surfer Gas Adsorption Porosimeter unit. Prior to the analysis, the fresh catalysts were pre-treated overnight under vacuum at 573 K. The surface structure and elemental composition of the solid samples were obtained from the

FESEM-EDX model JOEL/JSM-7800F. The powder X-ray diffraction (XRD) pattern of the calcined catalyst was acquired with a Rigaku Miniflex II unit using  $\text{Cu K}\alpha$  radiation ( $\lambda = 0.154 \text{ nm}$ ) operated at 15 mA and 30 kV. From the pattern, the crystallite size was calculated using the Scherer equation,  $d_{\text{crystallite}} = 0.94\lambda / (\phi \cos \theta)$ , where  $d_{\text{crystallite}}$  is crystallite size,  $\lambda$  is the wavelength of the radiation,  $\phi$  is the full-width at half maximum (FWHM) of the diffraction peak and  $\theta$  is the half of the diffraction angle.

### 2.3. Catalyst evaluation

Fig. 1 shows the experimental set up for the catalytic evaluation tests. The catalytic evaluation was carried out in a stainless steel fixed-bed reactor with 9.42 mm ID, 12.7 mm OD and a length of 400 mm that was positioned inside a split tube furnace. The catalyst bed, which comprised of 0.3 g of catalyst powder, was supported by quartz wool. The performance of each catalysts was evaluated from 1023 to 1173 K with various  $\text{CO}_2$ -to- $\text{CH}_4$  ratios ( $0.5 < \text{CMR} < 2.0$ ) under atmospheric pressure.  $\text{CH}_4$  and  $\text{CO}_2$  of purity >98% were supplied by Air Products and used as-received. All the inlet gas flow rates were regulated with an Alicat Scientific electronic mass flow controller (Model: MC-500SCCM-D). The exit gas was collected using 1-L Tedlar gas sampling bag, and the composition was determined by an online Agilent 19095P Gas Chromatography (GC) equipped with HP Plot Q column and HP Molecular Sieve column. The He gas was used as a carrier with flow rate of 20 ml/min, operating column temperature of 393 K and detector temperature of 423 K.

### 2.4. Reaction metrics

The catalyst performance was evaluated in terms of the methane conversion,  $X_{\text{CH}_4}$ , and the reaction rate,  $r$ .

The methane conversion to gaseous products is defined as:

$$X_{\text{CH}_4} = \frac{[F_{\text{CH}_4}]_{\text{in}} - [F_{\text{CH}_4}]_{\text{out}}}{[F_{\text{CH}_4}]_{\text{in}}} \times 100 \quad (2)$$

The rate of formation of gaseous compounds is given by:

$$r_i \left( \frac{\text{mol}}{\text{g}_{\text{cat}}\text{s}} \right) = \frac{y_i \times F_{\text{out}}}{W}; \quad i = \text{H}_2 \text{ and } \text{CO} \quad (3)$$

The rate of consumption of  $\text{CH}_4$  is:

$$r \left( \frac{\text{mol}}{\text{g}_{\text{cat}}\text{s}} \right) = \frac{F_{\text{in}} - (y_i \times F_{\text{out}})}{W} \quad (4)$$



**Table 1**  
Bulk composition of EFB clinker from XRF measurement.

Element	Composition (%)
Potassium (K)	13.93
Silicon (Si)	13.65
Calcium (Ca)	8.71
Iron (Fe)	4.10
Aluminum (Al)	1.76
Magnesium (Mg)	1.25
Phosphorus (P)	0.69
Sodium (Na)	0.44
Sulfur (S)	0.22
Titanium (Ti)	0.20
Manganese (Mn)	0.19
Strontium (Sr)	0.14
Barium (Ba)	0.06
Chlorine (Cl)	0.05
Rubidium (Rb)	0.05
Copper (Cu)	0.03
Chromium (Cr)	0.02
Nickel (Ni)	0.01
Zinc (Zn)	57 ppm
Cobalt (Co)	9 ppm

whereby  $F_{in}$  = inlet flow rate of  $CH_4$  and the  $y_i$  and  $F_{out}$  in both Eqs. (3) and (4) represent the dry basis molar composition and outlet flow rate from the reactor, respectively, while the  $W$  represents the weight of catalyst.

### 3. Results and discussion

#### 3.1. Catalyst properties

The crushed EFB clinker was analyzed for its bulk composition using XRF analysis. The results are as shown in Table 1. It can be seen from the XRF result that the main elements in the EFB clinker are potassium (K), silicon (Si) and calcium (Ca). Potassium and calcium elements, at compositions of 14.0% and 9.0%, respectively, are the main components that endowed the EFB clinker its alkaline characteristic. The other elements detected through this XRF analysis are either alkaline-based metals or transition metals. One of the important attributes of alkaline properties is its ability to soothe coke deposition during the hydrocarbon upgrading process, i.e. methane dry reforming, therefore justifying its use as a support for catalyst formulation in the current work. Furthermore, the EFB clinker is thermally-stable as it is a by-product of high temperature treatment for an extended period of time.

One of the selection criteria for determining suitability as a catalyst support is the material's thermal stability. Fig. 2 shows the thermal stability investigation via temperature-programmed calcination (TPC) whereby the derivative weight profiles of the pure EFB clinker, freshly-dried 20 wt% Co/80 wt% EFB clinker catalyst and freshly-dried 20 wt% Ni/80 wt% EFB clinker catalyst, at a ramping rate of 10 K/min were obtained under air-blanket. The thermal stability of the pure EFB clinker was proven by the flat profile obtained; this suggests that the EFB clinker can withstand temperatures up to 973 K (maximum temperature that can be handled by the TGA instrument) under air blanket without suffering any forms of thermal degradation. In addition, a single broad and distinctive calcination peak was recorded for each catalyst and the temperatures associated with these peaks hovered in between 420 K and 650 K with the peak maximum centered at an approximately 480 K. As these peaks were not observed for the pristine EFB clinker, therefore they were most likely an attribute of the  $Co(NO_3)_2$  and  $Ni(NO_3)_2$  decomposition into oxide metals, respectively.

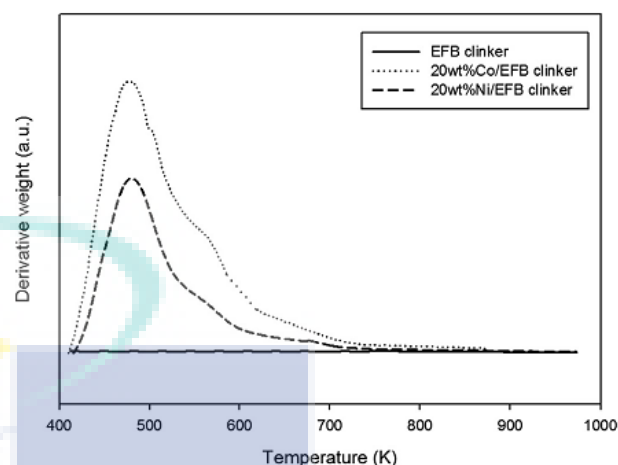


Fig. 2. Derivative weight profile obtained under air-blanket employing 10 K/min.

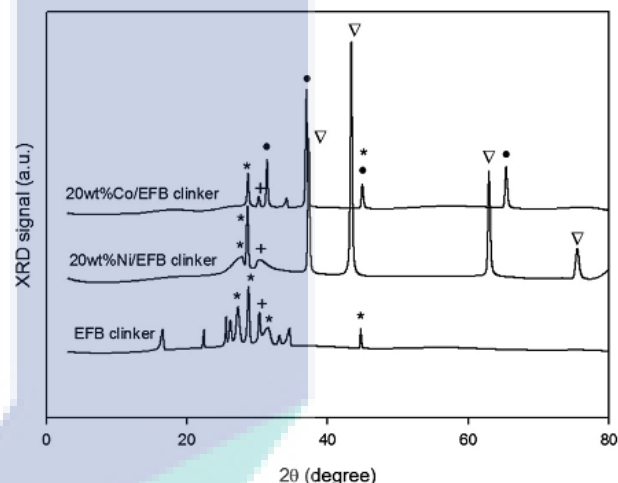


Fig. 3. XRD of EFB clinker, 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalysts, whereby (\*) is CaO, (+) is  $K_2O$ , ( $\nabla$ ) NiO, ( $\bullet$ )  $Co_2O_3$ .

The determination of the crystallinity phase of the oxide species was carried out using X-ray diffraction (XRD) technique. Fig. 3 shows the XRD spectrum of pristine EFB clinker as well as calcinated 20 wt% Ni/80 wt% EFB clinker catalyst and 20 wt% Co/80 wt% EFB clinker catalyst, respectively. Indeed, all the diffracted peaks were identified using the library provided by The International Centre for Diffraction Data (ICDD). For the EFB clinker, the peaks at around  $2\theta = 27.2^\circ$ ,  $28.7^\circ$  and  $31.5^\circ$ , represent the CaO species whilst  $K_2O$  species was recorded at  $30.5^\circ$ . In addition, it can also be observed that intensity of all the peaks was significantly lower than both the Co- and Ni-doped solid catalysts, respectively. Moreover, the peaks record appeared more diffuse than the as-prepared catalysts and also not as sharp as the Co- and Ni-catalysts. This indicates that the introduction of Co and Ni metals into the matrix of EFB clinker has improved the crystallinity of the prepared catalysts. Furthermore, some peaks that were originally present in the pristine EFB clinker were either merged or missing in the diffractogram of 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalysts (i.e.  $2\theta = 16.4^\circ$ ,  $22.3^\circ$ ,  $25.7^\circ$ ,  $26.0^\circ$ ,  $33.2^\circ$  and  $34.5^\circ$ ). This can be ascribed to the formation of new solid solution. For the 20 wt% Co/80 wt% EFB clinker catalyst, the XRD result shows peak at around  $2\theta = 28.7^\circ$ ,  $31.4^\circ$ ,  $37.0^\circ$ ,  $44.9^\circ$  and  $65.4^\circ$ . The

**Table 2**

Crystallite size determined for the peak around  $2\theta = 37.01^\circ$  for 20 wt% Co/80 wt% EFB clinker catalyst and  $2\theta = 43.38^\circ$  for 20 wt% Ni/80 wt% EFB clinker catalyst.

Sample	$2\theta$	Size (nm)
20 wt% Co/80 wt% EFB clinker catalyst	$37.0^\circ$	25
20 wt% Ni/80 wt% EFB clinker catalyst	$43.4^\circ$	21

peak at  $2\theta = 28.7^\circ$  represents CaO while the peaks at  $2\theta = 31.4^\circ$  and  $65.4^\circ$  represents  $\text{Co}_2\text{O}_3$ . For the 20 wt% Ni/80 wt% EFB clinker catalyst, the XRD spectra shows peak at around  $2\theta = 27.8^\circ$ ,  $30.2^\circ$ ,  $37.3^\circ$ ,  $43.4^\circ$ ,  $63.0^\circ$  and  $75.5^\circ$ . The peaks at  $2\theta = 27.8^\circ$  and  $30.2^\circ$  signify the presence of both CaO and  $\text{K}_2\text{O}$  species, while the peaks at  $2\theta = 37.3^\circ$ ,  $43.4^\circ$ ,  $63.0^\circ$  and  $75.5^\circ$  represent NiO. Moreover, it can be clearly seen that at around  $2\theta = 43.4^\circ$ , the peak has strong intensity indicating that the 20 wt% Ni/80 wt% EFB clinker has good crystallinity too. Peaks corresponding to NiO and  $\text{Co}_2\text{O}_3$  were observed for each catalyst due to high nickel and cobalt content and low nickel and cobalt dispersion in each catalyst. Table 2 shows the crystallite size of 20 wt% Co/80 wt% EFB clinker determined for the peak  $2\theta = 37.0^\circ$  and that of 20 wt% Ni/80 wt% EFB clinker

for the peak  $2\theta = 43.4$ . From Table 2, it can be observed that the crystallite size of 20 wt% Co/80 wt% EFB clinker catalyst (25 nm) and the 20 wt% Ni/80 wt% EFB clinker catalyst (21 nm) was almost invariant.

Fig. 4 shows the isotherm of the EFB clinker, 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalysts, respectively. The physisorption isotherm of pure EFB clinker was type IV isotherm according to the IUPAC system (cf. Fig. 4(a)) which was largely due to the mesoporous structure as apparent by the slight hysteresis that formed at  $P/P^0 \approx 1.0$ . The isotherms displayed a shift toward type V isotherm (mesoporous with weak interaction) upon Co and Ni metals introduction (cf. Figs. 4(b) and c). The hysteresis loops which represent capillary condensation effect, has grown more obvious, an indicative of increasing mesoporosity post metal addition. Significantly, this alteration was also confirmed by the values of BET specific surface area.

Table 3 shows that the entire samples exhibit relatively low BET specific surface area, in the range of  $2.50\text{--}3.90\text{ m}^2/\text{g}$ . It can be observed from Table 3 that the EFB clinker support itself was endowed with the lowest BET specific surface area ( $2.50\text{ m}^2/\text{g}$ ) among the three samples. Overall, the BET specific surface area showed mesoporosity structure for the entire samples consistent with the

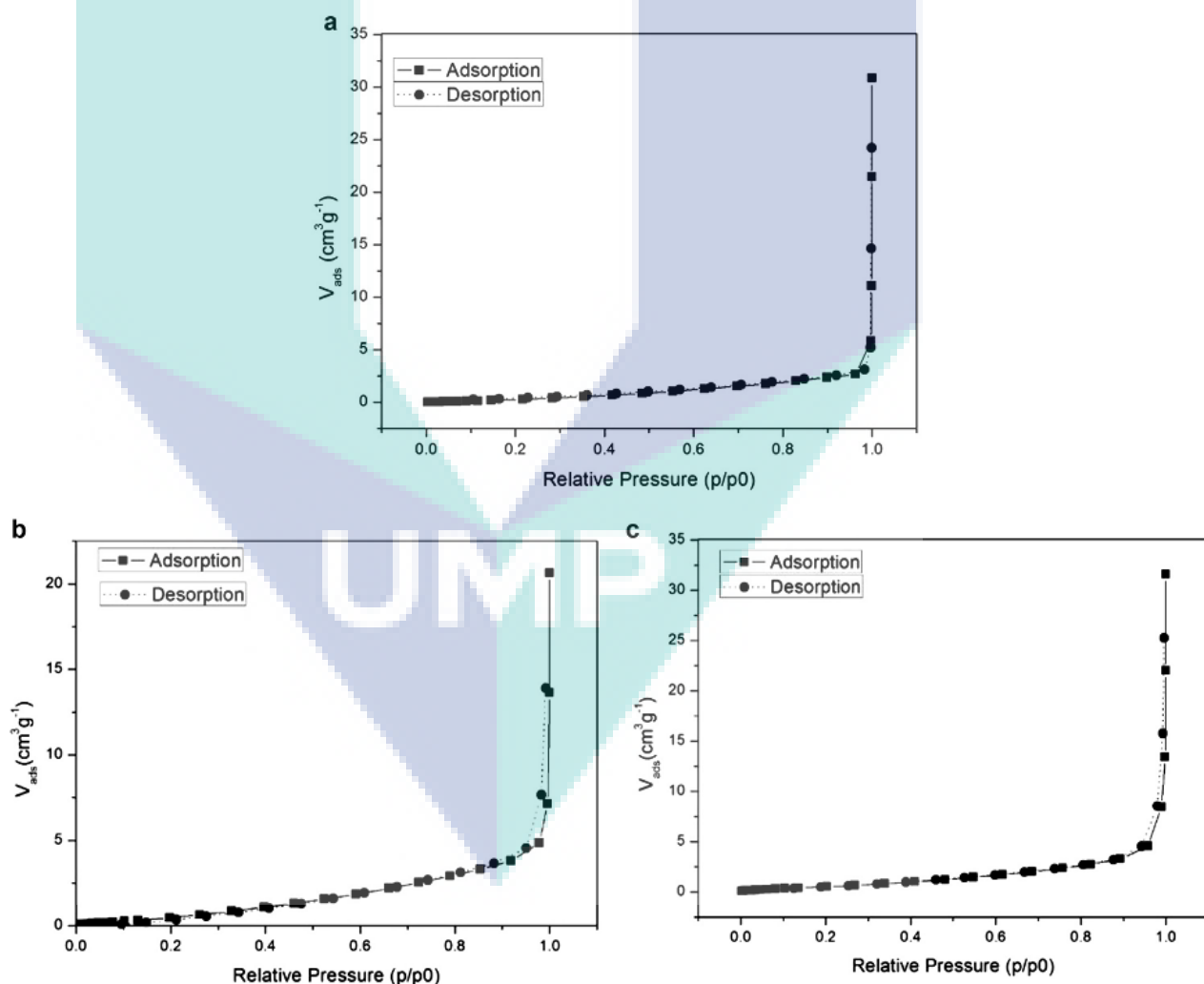


Fig. 4. Isotherm of (a) EFB clinker, (b) 20 wt% Co/80 wt% EFB clinker and (c) 20 wt% Ni/80 wt% EFB clinker catalysts.



**Table 3**  
BET specific surface area and pore volume of samples.

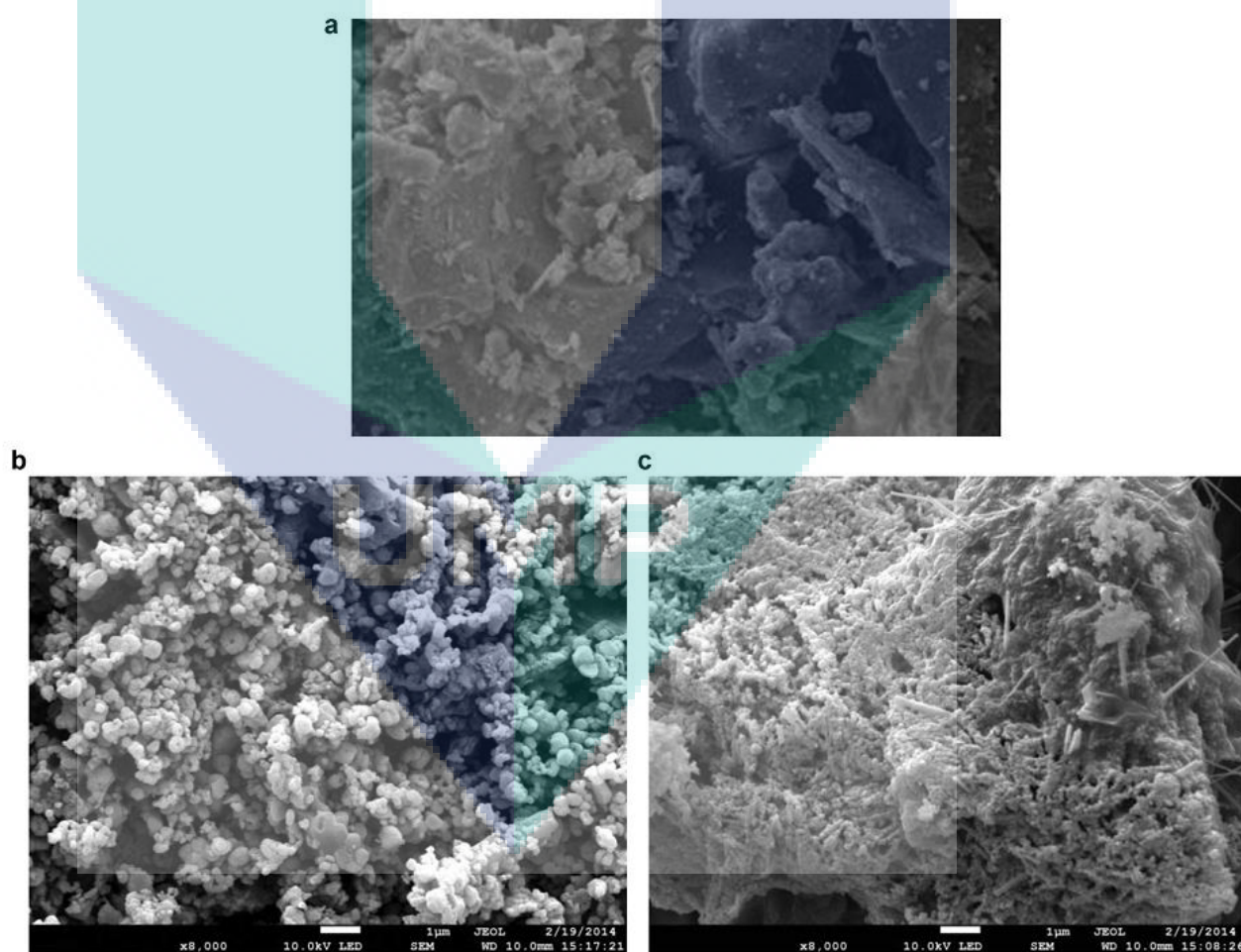
Sample	BET surface area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)
EFB clinker	2.48	0.0038
20 wt% Co/80 wt% EFB clinker	3.85	0.0055
20 wt% Ni/80 wt% EFB clinker	3.88	0.0069

isotherm plot in Fig. 4. Moreover, the BET specific surface area of the 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalysts (ca. 3.90 m<sup>2</sup>/g) showed an increment (50% magnitude) compared to the pristine EFB clinker. In addition, the cumulative pore volume also increased with the Co and Ni loading. These findings indicate that the addition of Co and Ni metals during the wet-impregnation procedure has successfully modified the morphology of the EFB clinker support, as evidenced by the N<sub>2</sub>-physorption isotherm and also corroborated by the FESEM images as shown in Fig. 5. It can be seen that the surface of pristine EFB clinker is smooth and has an irregular shape. In contrast, the surface of both 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker were rougher and bulkier than that of the pristine EFB clinker support which may be due to the deposition of Co<sub>2</sub>O<sub>3</sub> and NiO particles on the surface. The roughness attribute

has contributed to the increment in the mesoporosity of 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker catalysts as proven previously by the N<sub>2</sub>-physorption analysis.

### 3.2. Methane dry reforming

Fig. 6(a) shows the CH<sub>4</sub> conversion profiles at the 1st and 2nd h employing stoichiometric ratio of CO<sub>2</sub>:CH<sub>4</sub> at reaction temperature of 1173 K (highest temperature employed in the current investigation). The profiles showed that the conversion of CH<sub>4</sub> was relatively stable throughout the 2 h reaction duration with CH<sub>4</sub> conversion attaining near-80.0% for both 20 wt% Co/80 wt% EFB clinker and 20 wt% Ni/80 wt% EFB clinker. In comparison, this conversion level even has attained values reported for the most commonly-adopted Al<sub>2</sub>O<sub>3</sub>-support catalysts such as by Chen et al. [20] for 10 wt% Ni/90 wt% Al<sub>2</sub>O<sub>3</sub> catalyst (~80% CH<sub>4</sub> conversion at 1073 K) and also by Zeng et al. [21] for 20 wt% Co/80 wt% Al<sub>2</sub>O<sub>3</sub> catalyst (~80% CH<sub>4</sub> conversion at 1073 K). When the same reaction condition was imposed on the pure EFB clinker, no conversion of CH<sub>4</sub> was achieved and that the yield of syngas (H<sub>2</sub> and CO) was undetectable indicating that the role of EFB clinker was restricted to support only. In terms of H<sub>2</sub> and CO formation rates, Fig. 6(b) shows that the 20 wt% Ni/80 wt% EFB clinker catalyst yielded slightly higher CO formation rate ( $2.7 \times 10^{-6}$  mol/g<sub>cat</sub> s) compared to 20 wt% Co/80 wt% EFB clinker catalyst



**Fig. 5.** FESEM image of fresh (a) EFB clinker support, (b) 20 wt% Co/80 wt% EFB clinker and (c) 20 wt% Ni/80 wt% EFB clinker catalysts.

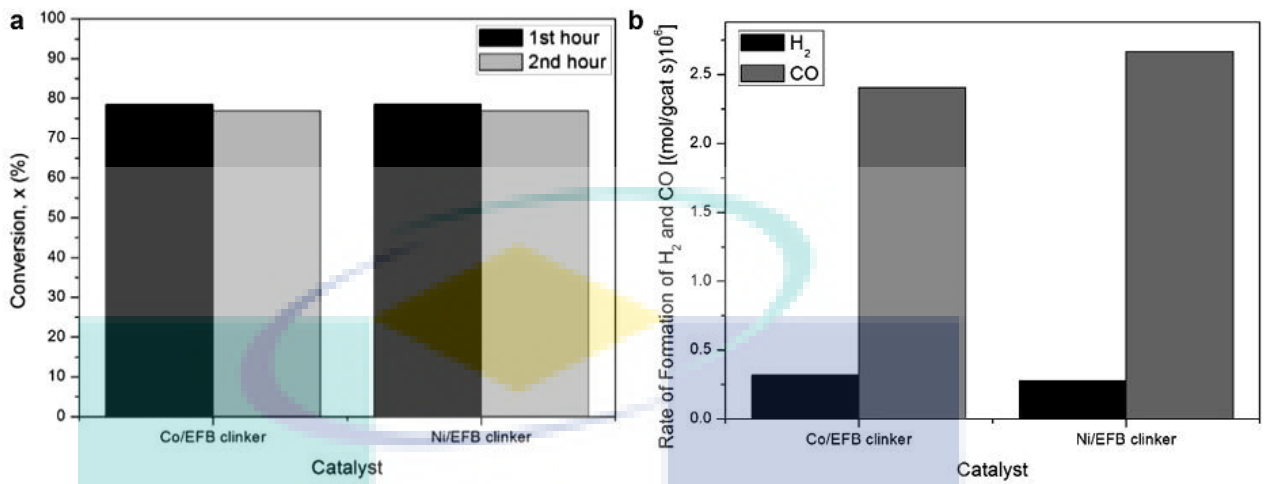


Fig. 6. (a) Transient profile of CH<sub>4</sub> conversion and (b) formation rates for both H<sub>2</sub> and CO ( $T = 1173\text{ K}$ ;  $\text{CO}_2:\text{CH}_4 = 1$ ;  $P = 1\text{ atm}$ ).

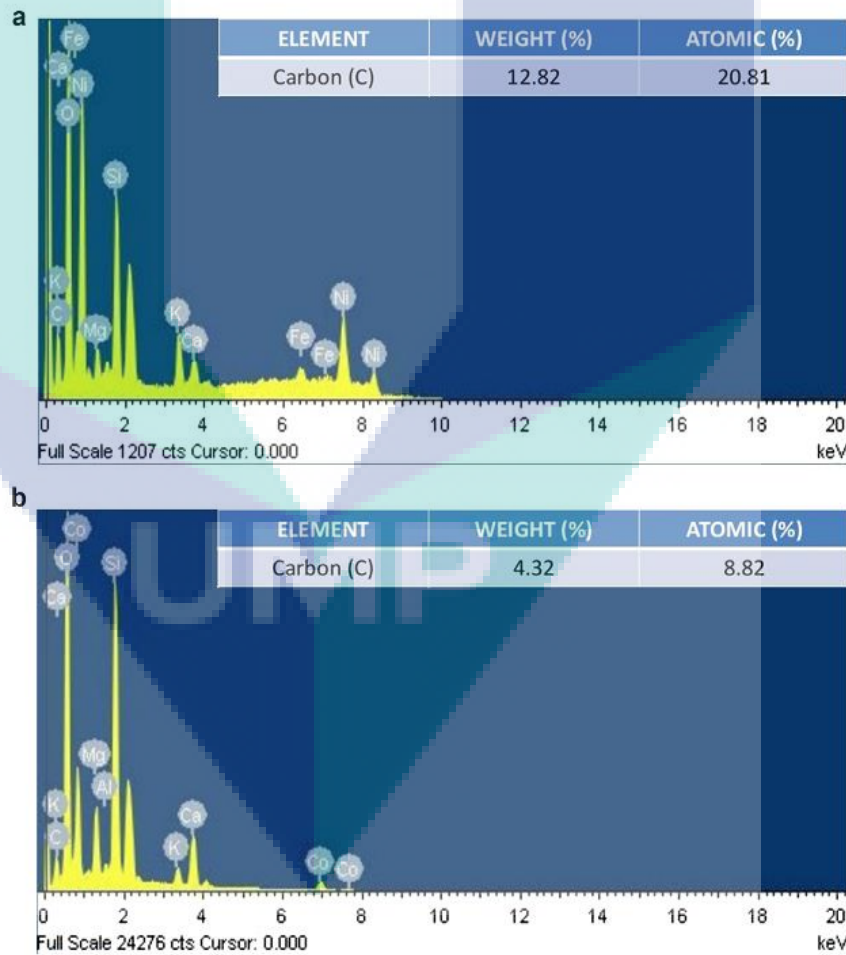


Fig. 7. EDX spectrum showing the amount of carbon element on (a) 20 wt% Ni/80 wt% EFB clinker and (b) 20 wt% Co/80 wt% EFB clinker catalysts.

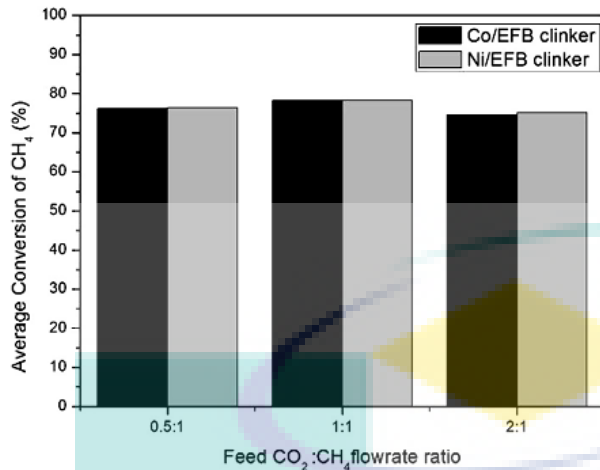
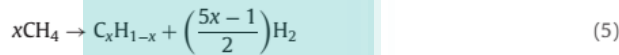
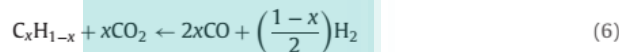


Fig. 8. Average conversion of CH<sub>4</sub> for various feed ratio ( $T = 1173$  K;  $P = 1$  atm).

( $2.4 \times 10^{-6}$  mol/g<sub>cat</sub> s). At the same time, the H<sub>2</sub> formation rates were almost similar for both types of catalysts ( $0.3 \times 10^{-6}$  mol/g<sub>cat</sub> s). According to Foo et al. [15], the methane dry reforming (cf. reaction in (1)) can be viewed as:



Subsequently, the interaction between CO<sub>2</sub> and the carbonaceous deposit yielded:



Under a significant presence of feed CO<sub>2</sub> concentration, any additional H<sub>2</sub> produced may be readily converted to CO and water via the reverse water-gas-shift reaction:



Reactions as outlined in Eqs. (5) to (7) may have explained the ratio of H<sub>2</sub>:CO which was lower than unity, i.e. CO formation rate was higher than H<sub>2</sub> formation rate. Significantly, if the main reaction (cf. Eq. (1)) was the sole occurring reaction, then in theory, the stoichiometry ratio of H<sub>2</sub>:CO should be 1.0. This indicates that Eq. (7) was very significant in the current reaction system.

In addition, the EDX analysis (cf. Fig. 7) that was subsequently taken of the used catalysts, after 2 h of reaction, showed that the carbon presence on the Ni metal (atomic % of 20.8) was significantly higher than the Co metal (atomic % of 8.8). This has confirmed that Ni metal is indeed more susceptible to carbon deposition than Co metal. According to Cheng et al. [18,19], Ni-based catalyst possessed higher acidity compared to Co-based catalyst, and that may have contributed to the higher carbon deposition over Ni-based catalysts in the current reaction.

Fig. 8 shows the conversion of CH<sub>4</sub> when CO<sub>2</sub>:CH<sub>4</sub> feed molar ratios were varied from 0.5 (under stoichiometric) to 2.0 (above stoichiometric) at 1173 K. For both sets of catalysts, as can be observed, the increase in the molar feed ratio from 0.5 to 1.0 has increased the average conversion of CH<sub>4</sub>. However, the conversion of CH<sub>4</sub> decreased when the reactant ratio was 2.0. Indeed, this observation is also corroborated with the results of previous investigations, referenced herein [15,22,23]. Under the stoichiometric ratio (CO<sub>2</sub>:CH<sub>4</sub> ratio of 0.5), there was an insufficient supply of CO<sub>2</sub> for converting CH<sub>4</sub>; hence a lower conversion of CH<sub>4</sub>. By increasing the CO<sub>2</sub> in the feed, the CH<sub>4</sub> conversion increased which validated the earlier proposition. Nevertheless, above the CO<sub>2</sub>:CH<sub>4</sub> ratio of 1.0, the CH<sub>4</sub> conversion dropped (cf. Fig. 8). This may be ascribed

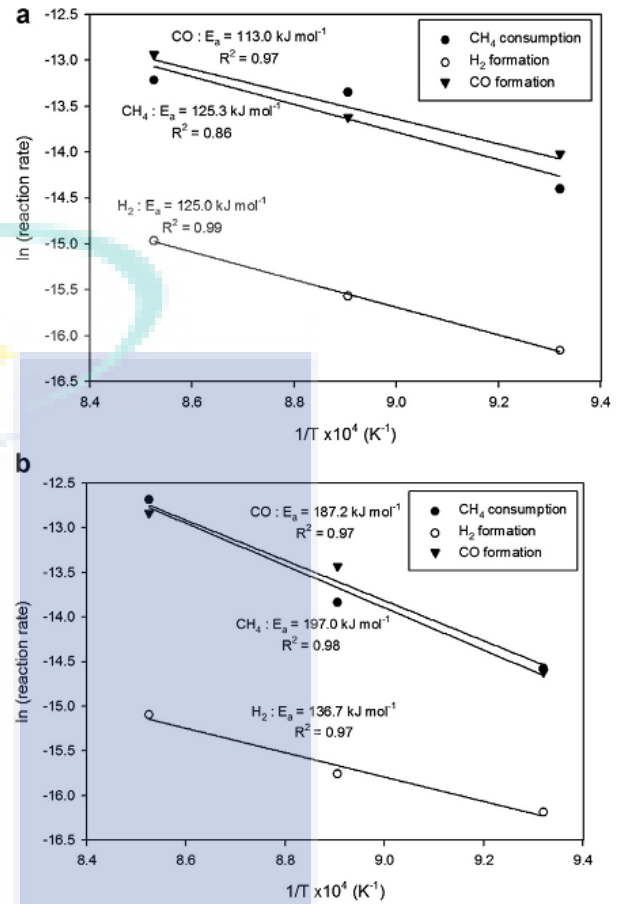


Fig. 9. Arrhenius plot of CH<sub>4</sub> consumption rate as well as formation rates of H<sub>2</sub> and CO at various temperatures for (a) 20 wt% Co/80 wt% EFB clinker and (b) 20 wt% Ni/80 wt% EFB clinker catalysts.

to the competing chemisorption of both CH<sub>4</sub> and CO<sub>2</sub> in the excess CO<sub>2</sub> environment that has hindered surface adsorption of CH<sub>4</sub>. Therefore, in this work, the maximum molar feed ratio to obtain the highest conversion was at CO<sub>2</sub>:CH<sub>4</sub> ratio of unity at 1773 K, which happens to correspond to its stoichiometric ratio.

In tandem with the Arrhenius principle, when the reforming temperature was increased, the reaction rate also increased. Graphs of  $\ln(r)$  versus  $1/T$  was plotted based on kinetic data of 20 wt% Ni/80 wt% EFB clinker catalyst and 20 wt% Co/80 wt% EFB clinker catalyst as shown in Fig. 9. From the slope of best fit, the activation energy was determined. For both sets of catalysts, the activation energy values were significantly higher than 100 kJ/mol, an important indication that the current reaction system was practically free from transport limitation (mass and heat transfers). Moreover, it can be seen that the  $E_a$  associated with CH<sub>4</sub> was 197.0 kJ/mol for 20 wt% Ni/EFB clinker catalyst whilst in contrast, it was 125.3 kJ/mol for 20 wt% Co/80 wt% EFB clinker catalyst. The discrepancy can be attributed to the unsatisfactory regression in particular for the Arrhenius fitting of 20 wt% Co/80 wt% EFB clinker catalyst. For the H<sub>2</sub> formation rate, both sets of catalysts yielded  $E_a$  values that were in close proximity to each other, at 125.0 (20 wt% Co 80 wt% EFB clinker) and 137.0 kJ/mol (20 wt% Ni/80 wt% EFB clinker), respectively. Nevertheless, for CO formation rates, 20 wt% Co/80 wt% EFB clinker catalyst yielded lower  $E_a$ , at 113.0 kJ/mol while it was 187.0 kJ/mol for the 20 wt% Ni/80 wt% EFB clinker catalyst.



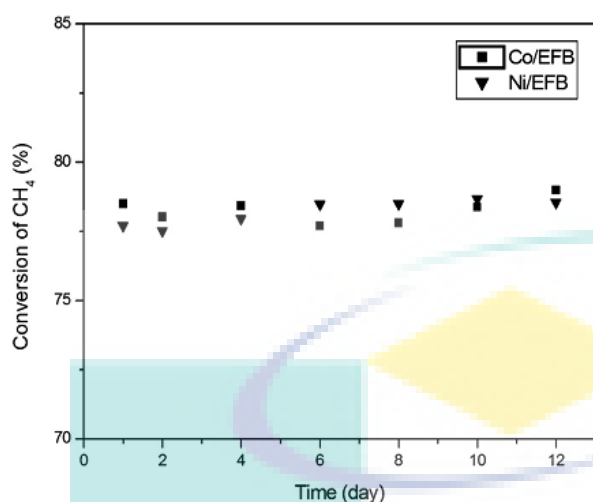


Fig. 10. Transient profiles of CH<sub>4</sub> conversion (%) over 12 days of time-on-stream.

Significantly, this has indicated that CO formation rate was faster over the Co-based catalyst than the Ni-based catalyst due to its lower activation energy. This showed that Co-catalyst was probably more effective in promoting the reverse water-gas-shift reaction (cf. Eq. (7)).

In order to study the catalytic longevity stability performance of both 20 wt% Co/80 wt% EFB clinker catalyst and 20 wt% Ni/80 wt% EFB clinker catalysts, experiments were conducted for 12 days with CH<sub>4</sub>:CO<sub>2</sub> ratio of 1.0 at 1173 K. The performance was probed in terms of stability of catalytic activity in particular conversion of CH<sub>4</sub> (cf. Fig. 10). Obviously, it shows that the conversion of CH<sub>4</sub> always remain in the range of 75–80% without any vigorous fluctuation throughout the 12 days reaction duration. Therefore, it can be inferred that both the 20 wt% Co/80 wt% EFB clinker catalyst and 20 wt% Ni/80 wt% EFB clinker catalyst did not exhibit any significant deactivation for 12 days of continuous reaction at 1173 K. The result obtained has demonstrated the excellent stability of both catalysts at the highest temperature employed in the current study. As aforementioned, EFB clinker is comprised of mainly basic elements such as K and Ca as summarized in Table 1. It is well known that carbon deposition is more severe on acidic-type of catalyst than basic catalyst, due to primarily a stronger “capturing” of carbon species to the acidic sites. In contrast, the presence of basic elements, due to its “electron-rich” nature, ensures that the chemical bonding between carbon (from both CH<sub>4</sub> and CO<sub>2</sub>) and catalyst is not overly strong to the point of fracturing the entire C–H bonds. Therefore, carbon deposition is less profound on basic catalyst, consequently, explains the more stable catalytic performance over prolonged reaction time. Hence, it can be conclusively confirmed that EFB clinker is suitable as support material due to its thermal stability as well as excellent CH<sub>4</sub> conversions (attaining 70.0–80.0%).

#### 4. Conclusions

In this study, we have determined that EFB clinker-supported Co and Ni catalysts were effective catalysts for methane dry reforming with high methane conversion (70–80% conversions), at CO<sub>2</sub>:CH<sub>4</sub> feed ratio of unity. The XRD of pristine EFB clinker showed two peaks that confirmed the presence of CaO and K<sub>2</sub>O. The calcined Co- and Ni-catalysts have XRD spectrum that showed strong intensity and good crystallinity for Co<sub>2</sub>O<sub>3</sub> and NiO species. The BET specific surface area of both Co/EFB clinker and Ni/EFB

clinker catalysts were larger compared to the pristine EFB clinker support. This finding indicates that the addition of Co and Ni metal during the wet-impregnation procedure successfully modified the surface of the EFB clinker support. The best conditions for syngas production via methane dry reforming were at CO<sub>2</sub>:CH<sub>4</sub> feed ratio of unity and 1173 K. Moreover, a study over 12 days of time-on-stream has indicated that the catalytic reforming performance was stable.

#### Acknowledgment

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DR. CHENG CHIN KUI  
Senior Lecturer.....  
Faculty of Chemical & Natural Resources Engineering  
Universiti Malaysia Pahang  
Lebuhraya Tun Razak, 26300 Gambang, Pahang.

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 Tajuk Projek : Synthesis and Characterization of EFB clinker Supported Nickel Catalyst for Syngas Production from Reactive Fluid Mixture of CO<sub>2</sub>-CH<sub>4</sub>  
 No. Vot Projek : RD140315  
 Fakulti : FKKSA  
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Tandatangan & Cop Ketua Penyelidik

DR. CHENG CHIN KUI  
 Senior Lecturer  
 Faculty of Chemical & Natural Resources Engineering  
 Universiti Malaysia Pahang  
 Lebuhraya Tun Razak, 26300 Gambang, Pahang  
 Disahkan Oleh Pengerusi J/Kuasa Penyelidikan Fakulti

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 PROFESOR DR. ABDURAHMAN HAMID NOUR  
 Timbalan Dekan (Penyelidikan & Pengejaian Siswazah)  
 Fakulti Kejuruteraan Kimia & Sumber Asli  
 Universiti Malaysia Pahang  
 Lebuhraya Tun Razak  
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PRELIMINARY IP SCREENING & TECHNOLOGY ASSESSMENT FORM

(To be completed by Project Leader submission of Final Report to RMC or whenever IP protection arrangement is required)

1. PROJECT TITLE IDENTIFICATION :

Synthesis and Characterization of EFB-clinker Supported Nickel Catalyst for  
Syngas Production from Reactive Fluid Mixture of  $\text{CO}_2$ - $\text{CH}_4$

Vote No: Rbu140315

2. PROJEK LEADER :

Name : CHENG CHIN KUI

Address : FKKSA, Universiti Malaysia Pahang

Tel : 095492896 Fax : 095492889 E-mail: chinkui@ump.edu.my

**3. DIRECT OUTPUT OF PROJECT** *(Please tick where applicable)*

Scientific Research	Applied Research	Product/ Process Development
<input type="checkbox"/> Algorithm	<input checked="" type="checkbox"/> Method/Technique	<input checked="" type="checkbox"/> Product/Component
<input type="checkbox"/> Structure	<input type="checkbox"/> Demonstration/Prototype	<input checked="" type="checkbox"/> Process
<input type="checkbox"/> Data	<input type="checkbox"/> Other, please Specify	<input type="checkbox"/> Software
<input type="checkbox"/> Other, please specify		<input type="checkbox"/> Other, please specify
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

**4. INTELLECTUAL PROPERTTY** *(Please tick where applicable)*

<input type="checkbox"/> Not patentable	<input type="checkbox"/> Inventor technology champion
<input checked="" type="checkbox"/> Patent search required	<input type="checkbox"/> Inventor team player
<input type="checkbox"/> Paten search completed and clean	<input type="checkbox"/> Monograph available
<input type="checkbox"/> Invention remains confidential	<input type="checkbox"/> Industrial partner identified
<input type="checkbox"/> No publications pending	<input type="checkbox"/> Patent Pending
<input type="checkbox"/> No prior claims to the technology	<input type="checkbox"/> Technology protected by patents pending

UMP

5. LIST OF EQUIPMENT BOUGHT USING THIS VOT

No	Item	Serial No	Location
	- Not Applicable -		

6. STATEMENT OF ACCOUNT

a)	APPROVED FUNDING	RM: <u>24900</u>
b)	TOTAL SPENDING	RM: <u>22 518.99</u>
c)	BALANCE	RM: <u>2 381.01</u>

## 7. TECHNICAL DESCRIPTION AND PERSPECTIVE

Please tick an executive summary of the new technology product, process, etc., describing how it works. Include brief analysis that competitive technology and signals the one that it may replace. Identify potential technology user group and the strategic means for exploitation.

a) **Technology Description**  
EFB clinker, a waste generated from incineration of empty fruit bunch has been employed as a support in the formulation of Ni/EFB catalyst for methane dry reforming. The reaction was successfully carried out with syngas being produced.

b) **Market Potential**  
Catalyst is a material that worths billion ringgit. Can be used in petrochemical industry.

c) **Commercialisation Strategies**  
Efforts on-going in applying for FRGS, followed by PRGS.

UMIP


8. RESEARCH PERFORMANCE EVALUATION

a) CHAIRMAN OF FACULTY RESEARCH COMMITTEE

Research Status	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Spending	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Overall Status	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	Excellent	Very Good	Good	Satisfactory	Fair	Weak

Comment/Recommendations :

The research status very good.  
- Research findings were published in a high impact factor journal (Scopus & ISI).

  
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Signature and stamp

Name : ABDURRAHMAN NOUR  
Date : 8/04/16

PROFESOR DR. ABDURRAHMAN HAMID NOUR  
Timbalan Dekan (Penyelidikan & Pengajian Siswazah)  
Fakulti Kejuruteraan Kimia & Sumber Asli  
Universiti Malaysia Pahang  
Labuhaya Tun Razak  
26300 Gambang, Kuantan,  
Pahang Darul Makmur  
Tel: 09-549 2922



**b) RMC EVALUATION**

Research Status	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Spending	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Overall Status	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
	Excellent	Very Good	Good	Satisfactory	Fair	Weak

**Comments :**

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**Recommendations :**

- Needs further research
- Patent application recommended
- Market without patent
- No tangible product. Report to be filed as reference

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Signature and Stamp of Director  
Research Management Centre

**Nama :** .....  
**Date :** .....