



Original Research Article

## Reverse water gas shift reaction over tungsten carbide prepared catalyst from waste date palm fronds at low temperatures reverse water gas shift reaction

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### ARTICLE INFORMATION

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### KEYWORDS

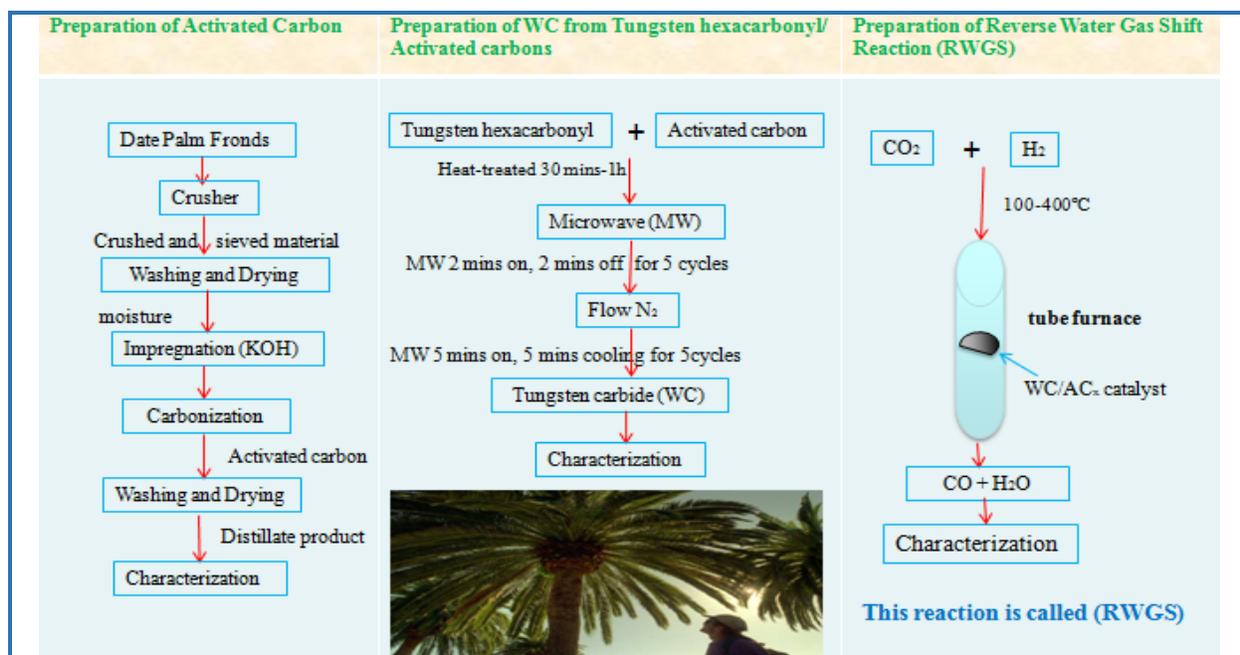
Activated carbon  
Potassium hydroxide  
Tungsten Carbide  
Reverse water gas shift

### ABSTRACT

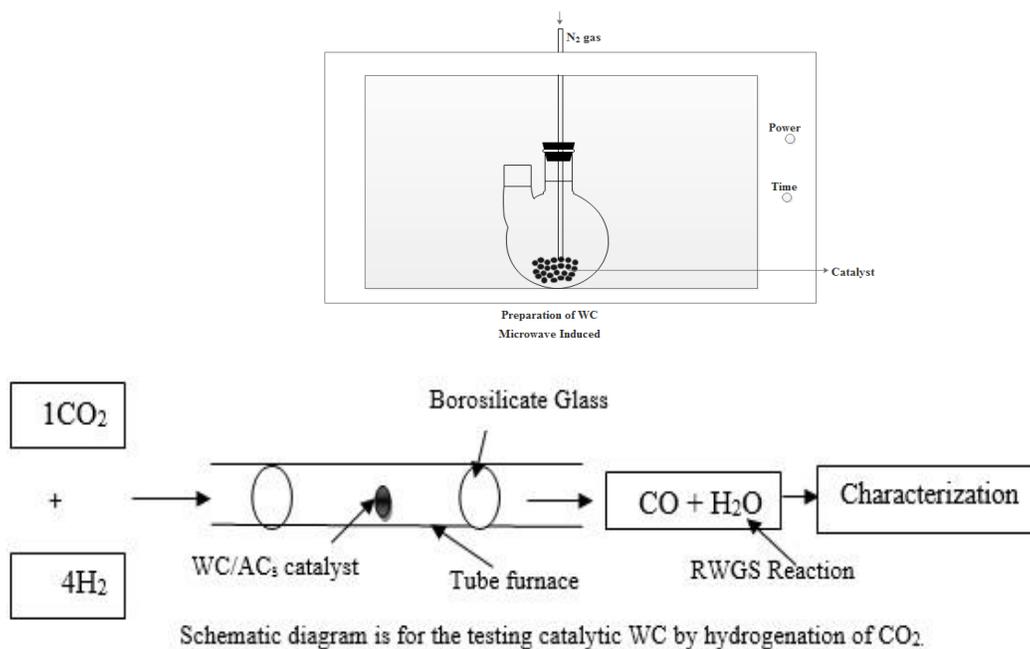
The reverse water gas shift reaction over the prepared tungsten carbide alloy (WC/AC) from date palm fronds catalyst was studied by CO<sub>2</sub> hydrogenation, temperature-programmed reduction of the WC/AC catalyst. In comparison to the reaction of CO<sub>2</sub> alone, hydrogen can significantly promote the CO formation in the RWGS reaction. The formate derived from association of H<sub>2</sub> and CO<sub>2</sub> is proposed to be the key intermediate for CO production. Formate dissociation mechanism is the major reaction route for CO production. The reverse water gas shift (RWGS) reaction over WC/AC with potassium (K) promoter was studied by means of CO<sub>2</sub> hydrogenation at temperature programmed. The main role of Potassium oxide (K<sub>2</sub>O) was to provide catalytic activity for decomposition of formates, besides acting as a promoter for CO<sub>2</sub> adsorption. Hydrogen was dissociatively adsorbed on WC/AC and could spill over to K<sub>2</sub>O to associate with CO<sub>2</sub>. This resulted in the formation of formate species for the production of CO.

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## Graphical Abstract



## Microwave Setup



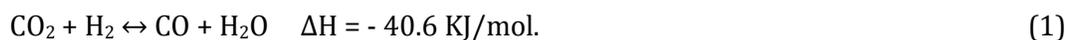
## Introduction

The green chemistry initiative arises from the need for sustainable development. The activities initiated by the chemists are primarily focused on large-scale processes, products and services that balance progress in quality of life and the preservation of human health and the environment [1]. For instance, activated carbon (AC) was applied to treat industrial wastewater and as catalyst or catalyst support due to characteristic absorption properties [2]. Waste materials are regarded as the most suitable source materials for the production of activated carbon because of their low purchasing cost and easy accessibility [3]. Additionally, generating activated carbons from a range of raw materials such as, agricultural wastes, coconut shells and wood will reduce the environmental impact of waste materials. At the same time, agricultural wastes have been used as activated carbon; a high added value can be obtained [4].

Currently, microwave power also plays a crucial part in adsorption capacity and carbon yield and microwave induced alloying. Increasing microwave power from 90 to 600 W considerably enhances yield, possibly because of the combined effect of internal and volumetric heating [5].

The reverse water gas shift (RWGS) reaction had been known to chemistry since the mid 1800's [6]. RWGS reaction was one of the most fundamental reactions which occur during supercritical water gasification [7, 8]. RWGS reaction is an equilibrium reaction and the conversion of CO<sub>2</sub> to CO is depending on temperature. Although high temperature reactions are effective for obtaining a high conversion, studies on the reactions at temperatures above 873 K are limited. The conversion of carbon dioxide to carbon monoxide by catalytic hydrogenation had been recognized as a very promising process. In industry, synthesis gas containing hydrogen and carbon monoxide can be used to prepare CH<sub>3</sub>OH as well as long chain hydrocarbons *via* the fischer-tropsch synthesis.

The water gas shift reaction (WGSR) which is an important industrial reaction for the production of chemicals or H<sub>2</sub>, have been expected to play a key role in the integration of gasification technologies with a hydrogen production/recovery unit [9]. The effluent stream of the gasifier, mainly hydrogen, carbon monoxide and carbon dioxide at high pressure reach to 30 atm and temperatures reach to 400-1000 °C [10, 11], would be directed to the water gas shift [12] reactor along with steam where reaction (Eq 1) will take place, increasing the yield of hydrogen [13] and [14].



This study aimed to prepare chemical activated carbons from date palm fronds waste using KOH as the activating agent. The best and optimized carbon was then selected for tungsten carbide microwave induce synthesis reactions. The main objective for this study is to synthesize tungsten

carbide using microwave induced from the prepared carbon and tungsten hexacarbonyl  $\{W(CO)_6\}$  as the precursor. The K moiety present in the carbon is expected to enhance both of the induced microwave alloying [7, 15].

## Experimental

### *Preparation of activated carbon (AC)*

In this study, the date palm fronds (DPF) were cut into small pieces of about 1-2 cm, washed with water and subsequently dried under the sun. These steps were followed by the crushing and sieving of the particles to mesh size of 1-4 mm. The palm pieces were then washed with hot water, and further subjected to heat from the sun to remove any moisture content. There after, the samples were wrapped in aluminum foil and carbonized at 400 °C for 2 hours in an evaporating dish at a heating rate of 30 °C/min. The resulting char was soaked in potassium hydroxide (KOH) solution with ratio of 1:1, 1:2, 1:3 (w/w%) respectively for 24 hours. The sample was then activated at power 360-630 W. The activated product was then cooled to room temperature and washed with 0.1 M hydrochloric acid and afterward with hot distilled water [7, 8].

### *Preparation of tungsten carbide*

In this study, the prepared carbon would be used in microwave induce alloying to the prepared tungsten carbide. The effect of KOH concentration was used in preparing the catalyst, tungsten hexacarbonyl powder was mixed with the appropriate amount of the activated carbon [6, 7].

There are several common methods used to prepare tungsten carbide catalyst and the one which was mostly used involved reaction of solid precursor, either as metal or oxide with gas phase containing reducing agent or carbonizing agent [9]. In this study, tungsten carbide catalyst was prepared using tungsten hexacarbonyl and activated carbon as precursors. In preparing the catalyst, tungsten hexacarbonyl powder  $[W(CO)_6]$  (1 g for all samples) was mixed with the appropriate activated carbons [AC (1:1), AC (1:2) and AC (1:3)]. The prepared sample was put together in glass Petri dish. Thus, glass Petri dish was placed on the hot plate, then set the ice bath on the lid and turn the heat on very low for 1 h, and to force the carbonyl to sublime evenly onto the activated carbon. Finally the catalyst was put heat treatment in the microwave induced with a power of 360-630 watt. The duration of microwave exposure was ranged from 2-5 mins under nitrogen gas. This activation takes about 4 hours, until remove all moisture of the catalyst. The tungsten carbide prepared was labelled as in Table 1.

### *Catalytic testing hydrogenation reaction of carbon dioxide*

**Table 1.** Experimental variables and sample codes for tungsten carbide supported on activated carbons

| Impregnation Ratio (in gram)                 | W(CO) <sub>6</sub> /ACs Activation Method |             |              |
|--|---|-------------|--------------|
|  | Energy (Watt)                             | Sample Code | Time (hours) |
| 1/1 [(1 g W(CO) <sub>6</sub> /1 g AC (1:1))] | 360-630                                   | WC (1:1)    | 4            |
| 1/2 [(1 g W(CO) <sub>6</sub> /2 g AC (1:2))] | 360-630                                   | WC (1:2)    | 4            |
| 1/3 [1 g W(CO) <sub>6</sub> /3 g AC (1:3)]   | 360-630                                   | WC (1:3)    | 4            |

Prior to the catalytic reaction, the prepared catalyst was reduced in hydrogen gas flow using a tube furnace. The catalyst was heated at 400 °C at the rate of 5 °C/min and hold at 400 °C for 1 hour. Then, about 1 g of the reduced catalyst was placed in the glass micro reactor. The hydrogenation reaction of carbon dioxide (H<sub>2</sub>/CO<sub>2</sub>) was conducted in a glass flow system at temperature ranging from 100 to 400 °C. However, the composition of the product evolved at the reactor outlet was collected in gas collector and were analyzed using Fourier transform infrared spectroscopy (FT-IR).

## Results and Discussion

### *Characterization of activated carbon and tungsten carbide*

#### *XRD analysis on tungsten carbide catalyst supported on ACs*

In this study, the effect of KOH activation ratio focus microwave induced alloy was recorded using XRD. Scanning  $2\theta$  from 10° to 90°, the WC film exhibits the following characteristic features:  $2\theta=30^\circ$ , WC 111;  $2\theta=31.6^\circ$ , WC 001;  $2\theta=35.8^\circ$ , WC 100;  $2\theta=48.4^\circ$ , WC 101;  $2\theta=64.8^\circ$  to  $65.2^\circ$ , WC 101;  $2\theta=73.4^\circ$ , WC 111 and  $2\theta=76.5-76.8^\circ$  WC 102. X-ray powder diffraction analysis of the WC catalysts prepared on activated carbon, and showed the formation of the W<sub>2</sub>C crystalline phase was characteristic features:  $2\theta=34.2^\circ$  and  $34.5^\circ$ , W<sub>2</sub>C 100;  $2\theta=39.8^\circ$ , W<sub>2</sub>C 101 during carbonization at 360 to 630 W only on carbon support as in [Figures 1, 2 and 3](#). However, it is possible, that on other carbon supports the W<sub>2</sub>C phase was very well dispersed; the W<sub>2</sub>C particles were too small to be detected by XRD.

[Figure 1](#) shows the XRD pattern of the WC (1:1). The peaks at the  $2\theta$  of 31.58°, 35.74°, 48.38°, 64.14°, 66.04°, 73.16°, 75.53°, 77.12°, and 84.16° are WC, the  $2\theta$  of 34.15° and 34.52° are W<sub>2</sub>C. [Figure 2](#) shows the XRD pattern of the WC (1:2). The peaks at the  $2\theta$  of 31.76°, 35.81°, 48.52°, 64.32°, 65.87°, 73.19°, 75.89°, 77.81°, and 84.56° are WC; the  $2\theta$  of 34.07° is W<sub>2</sub>C. As shown in [Figure 1](#), the peak of (002) planes at  $2\theta=24.9^\circ$ , WO<sub>3</sub> (002). In this result WC (1:2), there were no significant difference between WC (1:2) and the pattern WC (1:1), as it shows the difference only in the appearance of an

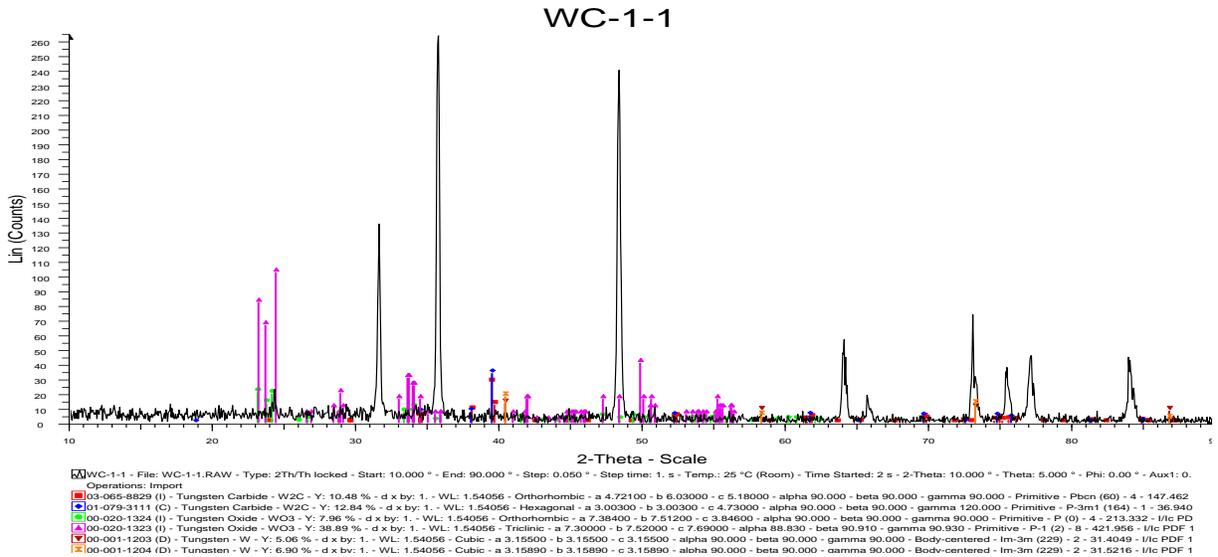


Figure 1. X-ray diffractogram of samples with WC (1:1) prepared

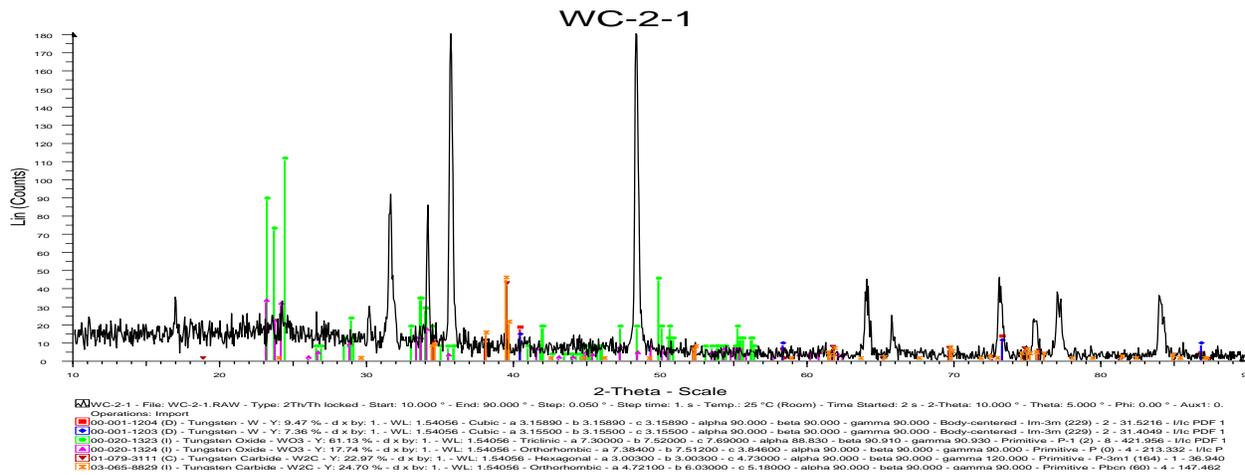


Figure 2. X-ray diffractogram of samples with WC (1:2) prepared

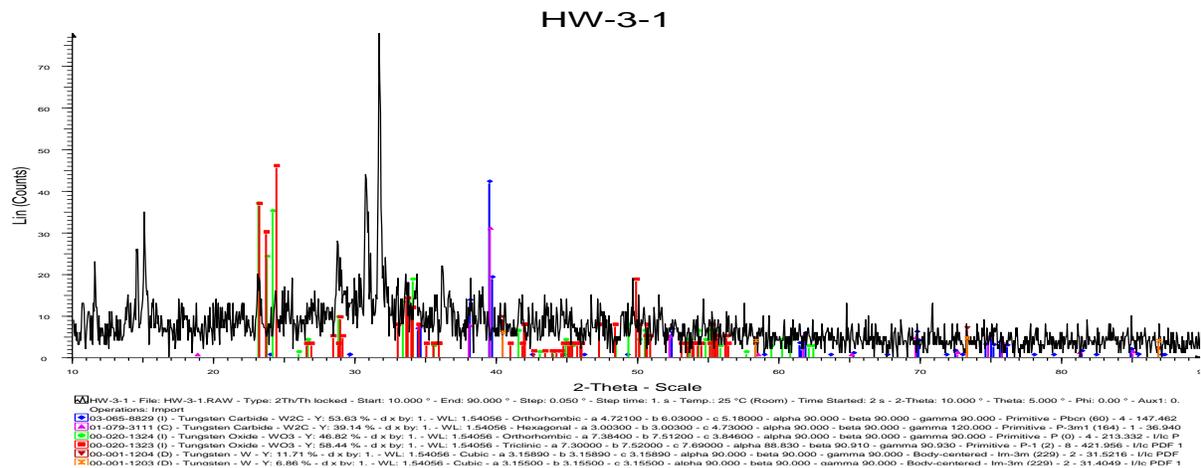
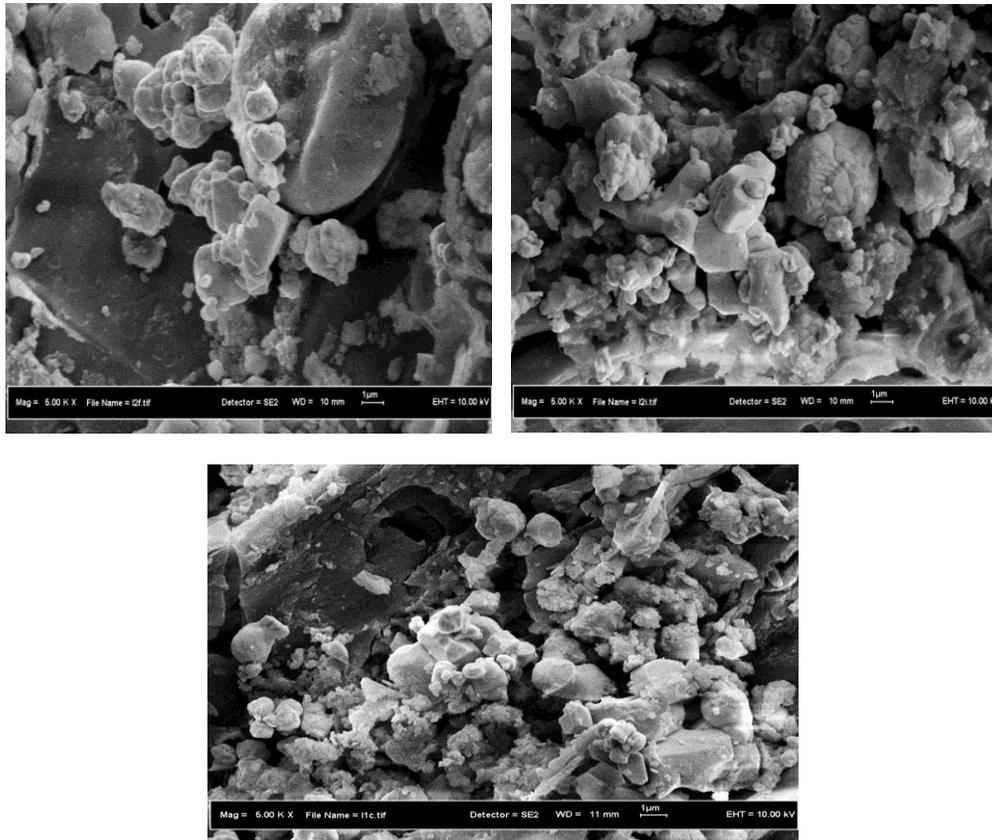


Figure 3. X-ray diffractogram of samples with WC (1:3) prepared

extra peak at the  $2\theta$  of  $17^\circ$  and  $24.5^\circ$  and corresponding to the (111) and (220) facets of  $WO_3$ , the  $2\theta$  of  $30.4^\circ$ ,  $31.6^\circ$ ,  $35.7^\circ$ ,  $48.4^\circ$ ,  $64.1^\circ$ - $65.9^\circ$ ,  $73.3^\circ$ ,  $75.9^\circ$ ,  $77.2^\circ$  and  $84.3^\circ$  and corresponding to the (0 1 1), (0 0 1), (1 0 0), (1 0 1), (1 0 1), (111), (201), (1 0 2) and (2 0 1) faces of WC. At  $2\theta$  of  $34.3^\circ$  are corresponding to the (1 0 0) face of  $W_2C$ . As shown in [Figure 3](#) gives the XRD patterns of the WC (1:3) unlike pattern of WC (1:1) and WC (1:2). There are some similar peaks of WC (1:1) and WC (1:2), and show some of peaks in various locations. The  $2\theta$  of  $31.7^\circ$ ,  $32.9^\circ$ ,  $35.7^\circ$ ,  $48.9^\circ$ ,  $64.8^\circ$ ,  $65.2^\circ$ ,  $73.4^\circ$ ,  $76.5^\circ$  and  $76.8^\circ$  and are corresponding to the (0 0 1), (2 0 2), (1 0 0), (1 0 1), (1 1 0), (0 0 2), (1 1 1), (1 0 2) and (2 0 1) faces of WC.

The X-ray diffractograms shown in [Figure 3](#) indicate the presence of both amorphous and crystalline materials. The main crystalline phase, identified by a high main peak at  $2\theta=31.66^\circ$  was hexagonal tungsten carbide (hexagonal-WC, [ $\alpha$ -WC]). The XRD patterns of the product obtained by  $W(CO)_6$  pyrolysis show the presence of hexagonal tungsten carbide ( $\alpha$ -WC), ([Figure 3](#)). From the XRD pattern in the sample WC (1:3), it is clear that the corresponding carbon peak is not observed, indicating the product consisting of disordered carbon. The Figure shows the XRD pattern of the product obtained by the pyrolysis of  $W(CO)_6$ . The carbon tubes (CNTs) produced by this method do not exhibit a nearly perfect nanotexture as those produced by the arc-discharge process. This observation is also further substantiating the results from the EDX studies. The formation of a carbon nanotube seems to require a suitable diameter of catalytic particles. Only when the diameter is smaller than a certain value, CNTs can be formed. This explains the results of EDX in the WC (1:3) as containing trace amounts of carbon while tungsten is very high. This means that the interaction of tungsten with carbon formed a high peak of hexagonal tungsten carbide ( $\alpha$ -WC). The results of XRD do not necessary show that the three peaks of tungsten carbide. There are some rare cases XRD results show one peak of tungsten carbide, as a result of conditions, the reaction mechanism and method of preparation. Moreover, some of previous and recent studies have shown the ability to see only one peak of tungsten carbide in the X-ray diffraction analysis.

[Figure 4](#) showed the micrograph of WC with magnification of 5.00 K $\times$ . The FESEM further confirmed the presence of WC and the secondary phases ( $W_2C$ ) already detected by XRD. Observations of the coatings using backscattered electron (BSE) imaging allowed the identification of a matrix phase which prepared WC from  $\{W(CO)_6\}$ . This WC phase of different composition was compared to the bulk material. The bright areas belong to the matrix areas with a high proportion of W (The element with the higher mean atomic number). The WC grains were located inside the splats, where the temperature attained is insufficient to dissolve the grains.  $W_2C$  was detected around the WC grains [[16](#)].



**Figure 4.** FESEM Micrograph of WC (1:1), WC (1:2) and WC (1:3)

#### *Energy dispersive X-ray spectroscopy (EDX) analysis*

The result on the surface composition of EDX for tungsten carbide prepared samples, which were shown in the elemental abundance, was listed in [Table 2](#). Elemental analysis was carried out to obtain the compositions of C, O, K, Si, Cl, Ca, and W atoms in the WC. As seen from [Table 2](#), element C was the most abundant constituent in WC (1:1), WC (1:2) and WC (1:3). With increased activation energy, there was an increasing in C content, probably due to the release of volatiles. The presence of K could also be explained from the addition of potassium hydroxide for activation process. Activation treatment has provoked a progressive release of oxygen, with a remarkable loss at energy 360-630 W. This implies that the fraction of oxygen was located around the pore entry, hence the extension of micropores at 360-630 watts resulted in a large decrease in oxygen, corresponding to a much higher C/O atomic ratio, as shown in WC (1:1) and WC (1:2).

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the pore entry, hence the extension of micropores at 360-630 watts resulted in a large decrease in oxygen, corresponding to a much higher C/O atomic ratio, as shown in WC (1:3). On the other hand, potassium hydroxide forms a condensation product in which the potassium ion gets attached to acidic functional group site, and water is removed from there during the formation of potassium salt of carboxylic acid as a condensation product. This revealed that KOH was strongly activated with precursor material constituent compounds.

This can be explained based on an assumption that CaO maybe have limited accessibility to the surface of date palm fronds (DPF) precursor because of its partial soluble nature in water. Moreover, it forms large amount of matrix with water soluble as well as surface constituent of DPF precursor that adherent to surface carbon. These formed stable calcium compounds on the carbon surface tend to fill interstice gaps between layers. After activation a small amount of this compound remained with the activated carbon. Perhaps this is why the presence of small amounts of calcium in the WC (1:1).

The larger Si-rich particles belong to date palm fronds, and therefore this may be another reason for the appearance of best result of WC (1:2) in XRD analysis. The WCs appeared completely covered by an irregular layer of deposit as shown in the [Figures 1, 2 and 3](#). A higher total carbon content in the material seems to decrease the amount of tungsten present in the three samples. This is indicated by the results from the EDX investigation. According to the results, the tungsten content had increased in the samples somewhat with increasing amount of total carbon. However, the mean value of several EDX analyses showed no significant variation at all. In accordance with the reasoning above concerning the amount of tungsten should also decrease with increasing carbon content. However, quantitative EDX of the binder phase shows that a lower overall carbon content increases the solubility of tungsten in the samples.

This is in good agreement with the results from the EDX analysis. The results can be understood from how the total carbon content in the material controls the amount of tungsten in the samples. The high oxygen content in EDX has led to the appearance of the high peaks in the XRD analysis. This may explain why the WC (1:3) contains few peaks of tungsten in XRD analysis.

**Table 2.** Measure EDX composition of tungsten carbide

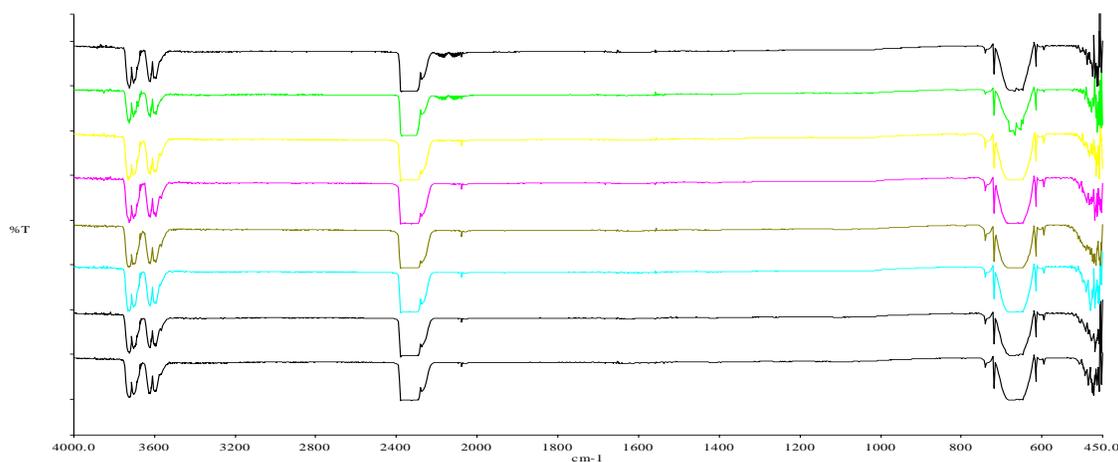
| Sample   | Intensity | Weight% |       |      |      |       |    |       |
|----------|-----------|---------|-------|------|------|-------|----|-------|
|          |           | C       | O     | K    | Ca   | Si    | Cl | W     |
| W (1:1)  | 0.64      | 59.36   | 29.49 | -    | 1.29 | 1.12  | -  | 8.74  |
| WC (1:2) | 0.72      | 33.3    | 30.36 | 1.89 | -    | 20.20 | -  | 14.24 |
| WC (1:3) | 0.94      | 11.21   | 7.67  | -    | -    | -     | -  | 81.12 |

Figure 5 displays a comparison of the FT-IR collected from a WC commercial sample and the prepared tungsten carbide catalyst (Figure 6). Both spectra exhibit most relevant features located at exactly the same vibrational frequencies, which indicates that both samples correspond to the same material, WC. In addition, the major product over WC commercial surface was the reverse water gas shift reaction (RWGS) ( $\text{CO}_2 + \text{H}_2 \leftrightarrow \text{CO} + \text{H}_2\text{O}$ ) which was produced by  $\text{CO}_2$  hydrogenation at programmed temperatures. In the case of WC commercial a substantial amount of RWGS was also produced. A strong dependence was showed the catalytic activity of WC commercial. This result shows that tungsten carbide can be excellent supports for enhancing the ability of transition metals to adsorb and activate  $\text{CO}_2/\text{H}_2$  toward the synthesis of RWGS.

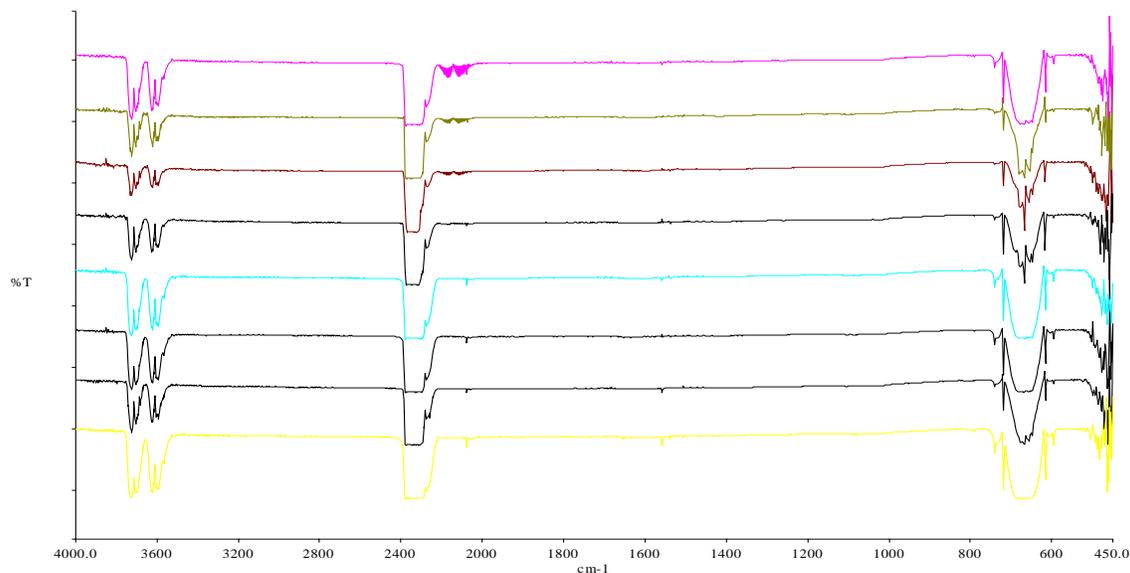
#### *FT-IR of $\text{CO}_2$ hydrogenation was using prepared WC*

The experimental data in Figures 6a and b and 7 indicate that WC(1:2) is as excellent catalysts for the production of CO (RWGS) from  $\text{CO}_2$  and using Converted  $\text{CO}_2$  calculations. Another route for the formation of CO (RWGS) from  $\text{CO}_2$  involves the reverse water gas shift reaction:  $\text{CO}_2 + \text{H}_2 \leftrightarrow \text{CO} + \text{H}_2\text{O}$ . Thus, the  $\text{CO}_2$  hydrogenation as a key intermediate, seems the best route for the production of the reverse water gas shift detected in the experiment for  $\text{CO}_2$  hydrogenation on WC(1:2) and WC commercial. The calculated results in the converted  $\text{CO}_2$  implies that the RWGS reaction was much fast, as observed experimentally.

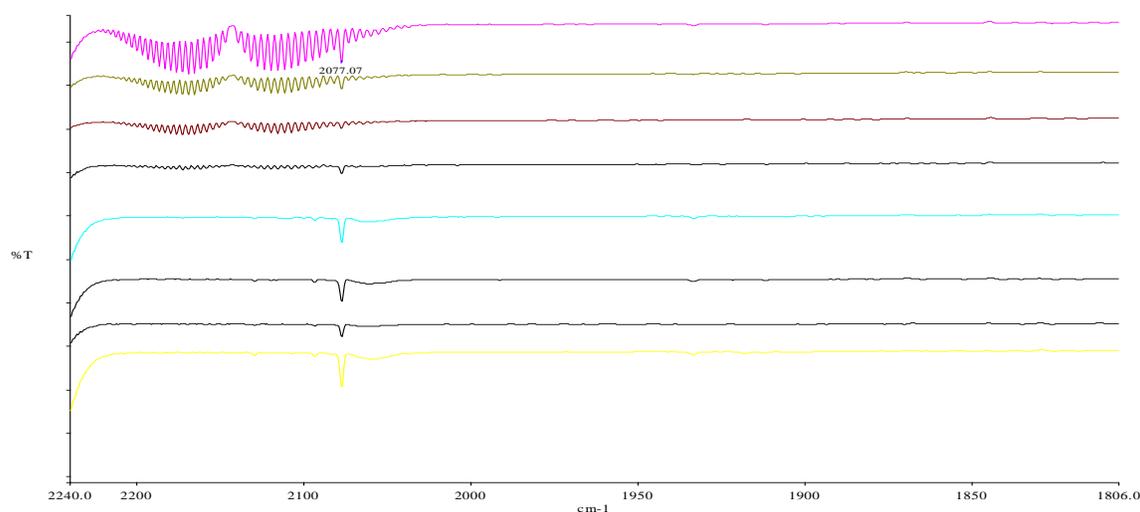
On other hand, Figures 6a, b and 7 display the effect of WC(1:2) and WC commercial on the production of RWGS. For  $\text{CO}_2/\text{H}_2$ , an increase was in WC(1:2) leads to a gradual decrease in the rate of production at 200 °C. in addition, the behavior for RWGS was different, with a increase in the production of this RWGS when going from a WC(1:2) at 250 to 350 °C. After reaching a maximum at this WC(1:2), the production of RWGS decreases with increasing temperature.



**Figure 5.** FT-IR spectra of  $\text{CO}_2$  hydrogenation over catalyst WC commercial at different temperatures



**Figure 6. a)** FT-IR spectra of CO<sub>2</sub> hydrogenation over catalyst WC (1:2) at different temperatures



**Figure 6. b)** FT-IR spectra of CO Spectrum enlarged WC (1:2) at different temperatures

A comparison of the data shown in [Figure 6a, b](#) and [7](#) investigated in this work indicates that WC deposition leads to the most active catalysts for formation of RWGS ( $\text{CO}_2 + \text{H}_2 \leftrightarrow \text{CO} + \text{H}_2\text{O}$ ). However, for the production of RWGS, WC(1:2) is the best by far. The converted CO<sub>2</sub> ratio is much larger on WC(1:2) than on WC commercial.

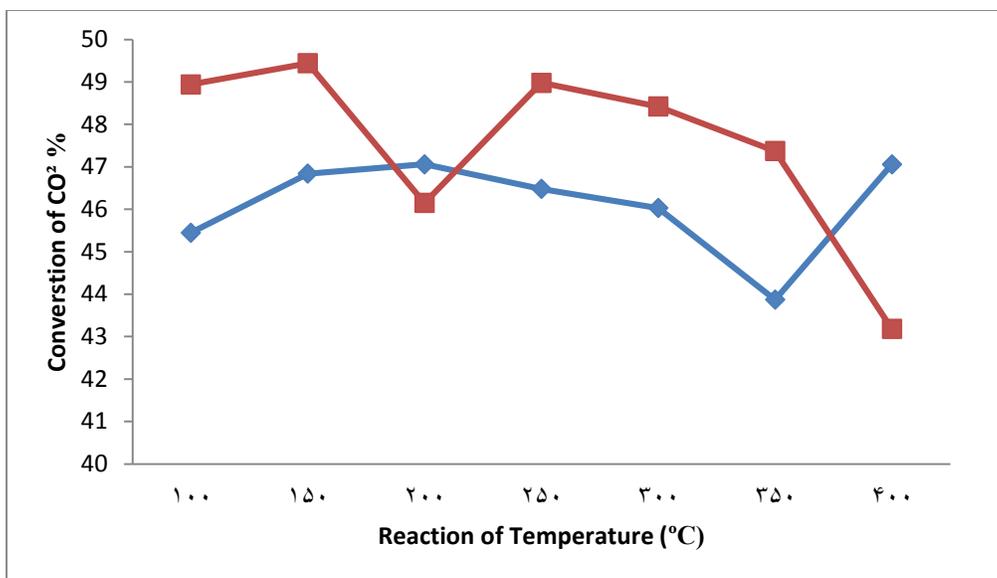
[Figure 7](#), the WC commercial and WC (1:2) catalysts show the best catalytic performance for CO<sub>2</sub> conversion. Further analysis of the spectra gave quantitative results for the species, as shown in [Figure 7](#) and [Table 3](#).

The effect of CO<sub>2</sub> hydrogenation on catalytic performance has been also compared to the WC commercial and WC (1:2) in order to investigate whether the beneficial effect of WC (1:2) on catalytic activity. The results presented in [Figure 7](#) show that WC (1:2) catalysts with WC commercial are practically active for CO<sub>2</sub> hydrogenation reactions and can mainly catalyze the shift of CO<sub>2</sub> to CO. This is in accordance to the results of our investigations, where it was shown that WC (1:2) catalyst exhibit exceptionally high activity for the RWGS reaction. However, WC (1:2) results in a significant increase of CO<sub>2</sub> conversion are accompanied by a shift of CO<sub>2</sub> conversion curve toward lower temperatures. Interestingly, the WC (1:2) catalyst is able to complete and CO<sub>2</sub> conversion to CO at temperatures as low as 100-400 °C. Thus, results obtained for both WC commercial and WC (1:2) catalyst which show hydrogenation of CO<sub>2</sub> are strongly favored to produce CO (RWGS reaction) and also are showed higher activity. Qualitatively similar results were obtained for all catalyst samples investigated here. Experimental results have shown that the nature of the support may play a crucial role in the mechanism of CO<sub>2</sub> hydrogenation reactions. In addition, catalytic performance of metal catalysts for hydrogenation reactions is depends on operating conditions used and metal support.

This study suggests that the prepared tungsten carbide gave higher catalytic activity which led to the conversion of CO<sub>2</sub> to carbon monoxide. This reaction has been known as reverse water gas reaction that is produced from hydrogenation of CO<sub>2</sub> process. Furthermore, comparing the WC (1:2) experimental results with the WC commercial, we observe the experimental results gave conversion of CO<sub>2</sub> into carbon monoxide better than WC commercial. Perhaps the reason is attributed to the use of activated carbon prepared from palm fronds that was used in the preparation of WC catalysts. We have systematically studied and compared tungsten carbide derived from preparation method as catalysts for the hydrogenation reaction. It can be employed as a reactant for the production of more complex molecules such as (Carbon monoxide) RWGS reaction, or methane or ammonia depending on temperatures used in the reaction.

In summary, activated carbon has been successfully synthesized and activated carbon has been used later as catalyst support for the preparation of tungsten carbide from tungsten hexacarbonyl by microwave induced approach. The excellent catalytic was performed in a simple preparation of the catalysts and used as alternative transition metal catalysts. In addition, the catalytic activity of this supported catalyst was investigated via hydrogenation CO<sub>2</sub> reactions. It is concluded that catalysts prepared WC/ACs show a better catalytic activity than tungsten commercial, which resulted in the production of RWGS reaction.

## Conclusion



**Figure 7.** Comparison of the hydrogenation reaction on the catalyst WC (1:2) and commercial

**Table 3.** Testing results of hydrogenation reaction over WC commercial and WC(1:2) catalysts at different temperatures

| Reaction Temperature |     | WC commercial                       | WC (1:2)                            |
|----------------------|-----|-------------------------------------|-------------------------------------|
| °C                   | K   | Converted CO <sub>2</sub> to CO (%) | Converted CO <sub>2</sub> to CO (%) |
| 100                  | 373 | 45.45                               | 48.94                               |
| 150                  | 423 | 46.84                               | 49.44                               |
| 200                  | 473 | 47.06                               | 46.15                               |
| 250                  | 523 | 46.48                               | 48.98                               |
| 300                  | 573 | 46.03                               | 48.42                               |
| 350                  | 623 | 43.87                               | 47.37                               |
| 400                  | 673 | 47.06                               | 43.18                               |

The catalytic activity of this supported catalyst was investigated via hydrogenation CO<sub>2</sub> reactions. It is concluded that catalysts which prepared WC (1:2) showed the best catalytic activity than tungsten commercial, which resulted in the production of reverse water gas shift (RWGS) reaction. RWGS rate was found to provide an improved description of the RWGS kinetic data. These results indicated that by decreasing the catalyst particle size, the activation energy of RWGS reaction increased and as a result, the reaction rate decreased consequently. The main role of K<sub>2</sub>O is to provide active sites for the formation of formates and adsorption of CO<sub>2</sub>. Hydrogen which was dissociatively adsorbed on WC (1:2) could be spilled over to K<sub>2</sub>O to associate with CO<sub>2</sub>, resulting in formation of

formate species. Potassium acts as a promoter for RWGS production on WC (1:2). The influence of CO on RWGS production from CO<sub>2</sub>+H<sub>2</sub> mixture on potassium-modified WC (1:2) was also investigated.

### Acknowledgements

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### Disclosure Statement

No potential conflict of interest was reported by the authors.

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