Influence of Noble Metal (Ru, Os and Ag) on the Reduction Behaviour of Iron Oxide Using Carbon Monoxide: TPR and Kinetic Studies

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Abstract—This study was undertaken to investigate the effect of noble metals (ruthenium, osmium and silver) on the reduction behaviour of iron oxide by (10%,v/v) carbon monoxide as a reductant. Powder of iron oxide samples doped with noble metal were prepared by impregnation method. The reduction behaviour of samples were characterized by temperature programmed reduction (TPR) and the phases formed of partially and completely reduced samples were characterized by X-ray diffraction spectroscopy (XRD). It is found that Ru enhanced the reducibility of the iron oxide compare to osmium and silver in the order as follows Ru-Fe₂O₃ > Os-Fe₂O₃ > Ag-Fe₂O₃. TPR results indicate that the reduction of Ru doped and undoped iron oxide proceed in three steps reduction (Fe₂O₃ \rightarrow Fe₃O₄ \rightarrow FeO \rightarrow Fe) with reduction to metal iron completed at lower temperature (650 °C) compared to undoped iron oxide (900 °C), while, Os-Fe₂O₃ and Ag-Fe₂O₃ inhibits complete reduction of Fe₂O₃ to metallic Fe by stabilizing the intermediate FeO as shown in the XRD profile. Furthermore, the decrease in the activation energy of Ru doped iron oxide regarding to all transition phases (Fe₂O₃ \rightarrow Fe₃O₄ \rightarrow $FeO \rightarrow Fe$) during the reduction process may also led to the increase in the rates of iron oxide reduction.

Index Terms—Reduction, iron oxide, noble metal, carbon monoxide.

I. INTRODUCTION

The reduction of Fe₂O₃ was a subject of numerous studies and generally used as an adsorbent, a catalyst precursor and an active component of catalytic material [1]. As a catalyst, the reduction behavior and the degree of reduction of the iron species were highly important in such application in industry and some chemical processes [1], [2]. According to other researchers [3] reducibility is one of the tools to studies the interactions between the metal and the support material in supported catalytic systems. The reduction behavior of Fe₂O₃ is greatly affected by various factors such as temperature, concentration reduction gas used, particle size, crystallinity and additives to the $Fe_2O_3[1]$, [4]-[6]. The reduction of iron oxide is a complex gas-solid reaction influenced by a complicated structural changes in the intermediate oxides which may include several steps and possibilities indicating two-step mechanism i.e. $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow Fe$ or the three-step reaction, i.e. $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow FeO \rightarrow Fe$ which the reaction usually simultaneous [7], [8]. Other researcher [3] proposed the iron oxide reduction routes depending on the reduction temperature are as following:

$$Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow Fe$$
for T<450 °C $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow Fe_xO + Fe \rightarrow Fe$ for 450 °C $Fe_2O_3 \rightarrow Fe_3O_4 \rightarrow FeO \rightarrow Fe$ for T>570 °C

Furthermore, noble metal as an additives in particular are more active, stable and can be obtained in a high degree of dispersion on the metal oxide support which allows to improve the catalytic activity of some chemical processes [9], [10]. To enhance the reduction performance of iron oxide, the addition of noble metals e.g. (Ru, Os and Ag) as additives was considered. Previously, studies of the reduction of doped iron oxide catalyst somehow mostly focused on characterization issues rather than on kinetics simply because of the complexity of the reduction process. Thus, in this work, the nonisothermal temperature programmed reduction was employed to investigate the reduction behavior of Ru, Os and Ag doped iron oxide and compare it to none doped species. In addition, the best performance of doped iron catalyst will be selected for the kinetic studies which will be carried out using different heating rate.

II. METHODOLOGY

A. Materials

Iron (III) oxide (99%) was obtained from Sigma Aldrich Chemical Company and Ruthenium (III) Chloride (99.9%) from Acros Organic, Osmium (III) Chloride hydrate from Sigma Aldrich, silver nitrate from Systerm, Gas mixture of carbon monoxide in nitrogen (10% CO) was obtained from MOX.

B. Catalyst Preparation

The Ru, Os, Ag doped to iron oxide was prepared by impregnating Fe_2O_3 powder with aqueous solution containing cation additives. The amount of ruthenium, osmium and silver added was adjust to be 3 (mol %) of total metal cation. The Fe_2O_3 powder was directly impregnated into 50 ml distilled water corresponding metal cation additives in the above proportion and stirred vigorously for 5 hours at room temperature. The impregnated sample was dried at 110 °C overnight and subsequently calcined at 600 °C for 3 hours. The Fe_2O_3 sample with ruthenium, osmium, silver and without denoted as Ru-Fe_2O_3, Os-Fe_2O_3, Ag-Fe_2O_3 and Fe_2O_3, respectively.

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C. Analysis and Characterization

Temperature programmed reduction (TPR) measurements using a Micromeritic Autochem 2920 Chemisorption Analyzer apparatus; a feed of 10% (v/v) CO in N₂ at a flow rate of 20 mL min⁻¹ (STP) was used as a reducing gas. The 50 mg of samples were heated up to 900 °C, at a rate of 10 °C min^{-1} and for the kinetic study, different heating rate (10, 13, $15 \, {}^{0}\text{C} \text{ min}^{-1}$) were applied to the samples. The carbon monoxide consumption was monitored using a thermal conductivity detector (TCD). Phase characterization was carried out by X-ray diffraction (XRD) Bruker AXS D8 Advance type with X-ray radiation source of Cu Ka (40 kV, 40 mA) to record the 20 diffraction angle from 10-80 degree at wavelength ($\lambda = 0.154$ nm) to observe the lattice of the structures. For identification purposes of crystalline phase composition, diffraction patterns obtained were matched with standard diffraction (JCPDS) files. Physical surface analysis (BET) of nitrogen adsorption at 77 K (liquid nitrogen) was conducted using a Micromeritics ASAP 2010 instrument to obtain the adsorption isotherm of each sample. The Brunauer-Emmett-Teller (BET) surface area was also calculated from the isotherms. Before analyses were done, samples were degassed at 350 °C for 6 h.

III. RESULTS AND DISCUSSION

A. Structural Properties by XRD

Fig. 1 shows the X-ray diffraction patterns of prepared samples of Ru-Fe₂O₃, Os-Fe₂O₃, Ag-Fe₂O₃ and undoped Fe_2O_3 before reduction with CO in nitrogen (10%, v/v). The characteristic peaks are consistent with the standard data of rhombohedral (α -Fe₂O₃, JCPDS 33-0664) with the 2 θ Fe₂O₃ main peaks reveal the presence of contribution at about 24.2, 33.2, 35.7, 40.9, 49.5, 53.5, 57.2, 62.4, 64.2, 72.4 and 75.7. It should be noticed that addition of respective noble metals give the diffraction peaks shifted to higher 2θ positions which is mainly due to the lattice constant has changed with the interference of the doped element. Furthermore, the diffraction peaks showing improvement in the crystallinity when loading with Ru, Os and Ag but any diffraction peaks ascribed to their oxides were not observed for the samples containing these additives. These observations suggest a high dispersion of the particles loaded on the iron oxide, a formation of non-crystalline compounds or compounds giving the same diffraction peaks as those of Fe_2O_3 [11].

To investigate the role of Ru, Os and Ag in the reduction behaviour of Fe_2O_3 , the phases formed after the reduction were analyzed by recording the XRD pattern of the residuals obtained after TPR experiments. The samples were collected according to the TPR profile when the reduction of doped Fe_2O_3 are assumed to be finished. Meanwhile, undoped Fe_2O_3 was also collected at related temperature for the comparison purposes.

For Ru-Fe₂O₃, the reduction process is assumed to be completed when the temperature reached 650 $^{\circ}$ C. The XRD diffractograms of the Ru-Fe₂O₃ and undoped Fe₂O₃ at temperature 650 $^{\circ}$ C and 900 $^{\circ}$ C are shown in Fig. 2. The results show Ru-Fe₂O₃ only gives a high degree of crystallinity of metallic Fe diffraction peak (iron, JCPDS 65-4899) at temperature 650 0 C while undoped Fe₂O₃ still consist of the remaining crystallite phases of cubic Fe₃O₄ (3,1,1), Fe₃O₄ (4,4,0) (magnetite, JCPDS 71-6336) and crystallite phases of cubic FeO (1,1,1), FeO (2,0,0), FeO (2,2,0), FeO (3,1,1) and FeO (2,2,2) (wustite, JCPDS 80-0686).



Fig. 1. XRD diffractogram of (a) Fe₂O₃, (b) Ru-Fe₂O₃, (c) Os-Fe₂O₃ and (d) Ag-Fe₂O₃ calcined at 600 ^{0}C . () Fe₂O₃.



Fig. 2. XRD diffractogram of (a) Fe_2O_3 and (b) Ru- Fe_2O_3 after reduction at 650 °C, (c) Fe_2O_3 and (d) Ru- Fe_2O_3 after reduction at 900 °C. (•) Fe_3O_4 , (•) FeO and (•) metallic Fe.

Meanwhile, XRD patterns for Os-Fe₂O₃ and undoped Fe₂O₃ which was recorded after reduction at temperature 700 °C and 900 °C are shown in Fig. 3. From the results, at reduction temperature of 700 °C, Os-Fe₂O₃ display better reducibility compare to the undoped Fe₂O₃. At this temperature, the undoped Fe₂O₃ shows a trace of intermediate Fe₃O₄ which was not detected in Os-Fe₂O₃. However, as the temperature increase to 900 °C, the XRD pattern of Os-Fe₂O₃ still exhibited a small remaining unreduced FeO characteristic peaks at $2\theta = 42.1^{\circ}$ and 60.9. Contrary with Fe₂O₃ with no other phases related to the iron oxide intermediate indicates a better reduction ability with the entire reduction to metallic Fe has completed within 900 °C.

Moreover, XRD pattern for Ag-Fe₂O₃ are given in Fig. 4. Different pattern was observed for Ag-Fe₂O₃ which the reduction ability is rather lower than the undoped Fe₂O₃. Result shows at temperature 650 0 C, a mixture of Fe₃O₄ and FeO diffraction peaks were observed with absent of metallic Fe. There is also a small trace of Ag metal with crystallite phases of Ag (1,1,1) follow (silver, JCPDS 65-2871) for both

temperature 650 $^{\circ}$ C and 900 $^{\circ}$ C. Furthermore, as the reduction temperature reached 900 $^{\circ}$ C, the XRD results still show a characteristic of intermediate FeO but somehow were not detected in undoped Fe₂O₃. It is found that Ag-Fe₂O₃ inhibits complete reduction to metallic Fe by stabilizing the intermediate FeO.



Fig. 3. XRD diffractogram of (a) Fe_2O_3 and (b) Os-Fe_2O_3 after reduction at 700 °C, (c) Fe_2O_3 and (d) Os-Fe_2O_3 after reduction at 900 °C. (\bullet) Fe_3O_4 , (\diamond) FeO and (\blacksquare) metallic Fe.



Fig. 4. XRD diffractogram of (a) Fe_2O_3 and (b) Ag-Fe_2O_3 after reduction at 650 °C and (c) Fe_2O_3 and (d) Ag-Fe_2O_3 after reduction at 900 °C. (\bullet) Fe_3O_4 , (\bullet) FeO, (\bullet) metallic Fe and Ag (\star).

B. Structural Properties by Isotherm Adsorption of N_2

Table I summarizes the BET surface areas of the prepared Ru-Fe₂O₃, Os-Fe₂O₃, Ag-Fe₂O₃ and undoped Fe₂O₃. The result indicates that Ru-Fe₂O₃ (5.27 m²g) and Os-Fe₂O₃ (4.89 m²g), show the BET surface area were larger than undoped Fe₂O₃ (4.57 m²g) while Ag-Fe₂O₃ give the lowest value among all. Moreover, Hu, Gao, & Yang, 2007 [12] mentioned that specific surface area usually results in more unsaturated surface coordination sites exposed to the gas which may result in the increasing of the reducibility performance of the iron oxide in this study.

TABLE I: STRUCTURAL PROPERTIES

Sample	BET surface area	
	m²/g	
Ru-Fe ₂ O ₃	5.27	
Os-Fe ₂ O ₃	4.89	
Ag-Fe ₂ O ₃	3.86	
Fe ₂ O ₃	4.57	

C. Chemical Properties by TPR Analysis

The reduction properties of all samples were determined by the TPR experiment. The TPR profiles of prepared Ru-Fe₂O₃, Os-Fe₂O₃, Ag-Fe₂O₃ and undoped Fe₂O₃ in nonisothermal reduction under 10% CO in nitrogen are shown in Fig. 5.

TPR profiles of undoped Fe₂O₃ displayed a 3 steps reduction mechanism with one reduction peak at lower temperature (361 ⁰C) represent the reduction of $Fe_2O_3 \rightarrow$ Fe₃O₄ and 2 broad peaks at 509 °C and 807 °C are subsequent to reduction steps of $Fe_3O_4 \rightarrow FeO$ and $FeO \rightarrow$ metallic Fe. Whereas, addition of noble metals to the Fe₂O₃ give different information about the reducibility of Fe₂O₃. TPR results show that the interaction between noble metals Ru and Os to the Fe₂O₃, respectively, decreased the reduction temperature and all peaks are shifted to lower temperature. Doping with these metals generate the amount of oxygen released, and lowers the TPR reduction temperature. While Ag doped Fe₂O₃ shows a very small peak at 620 ^oC but no improvement on the reduction ability compare to undoped Fe₂O₃ and only partial reduction occur when the temperature reached 900 °C as coincides with the appearance of crystalline FeO observed in the XRD experiments. Thus, higher temperature is requires for the deep reduction of metallic Fe to be achieved due to the strong interaction of Ag element with Fe₂O₃.

However only Ru-Fe₂O₃ enhance the reducibility of the iron oxide to metallic Fe by completed at 650 °C which was lower than undoped $Fe_2O_3(900 \ ^0C)$ as also confirmed by the XRD analysis. 3 reduction events of Ru-Fe₂O₃ reflected by 3 peaks around 319 °C, 475 °C and 567 °C also showing a stepwise of Fe₂O₃ to Fe through Fe₃O₄ and FeO as an intermediate. Meanwhile for Os-Fe₂O₃, the temperature for the first peak obtained is 329 °C and for higher temperature, the peaks were observed overlaping to each other with a shoulder of 567 °C and a peak at 591 °C. Even though the TPR pattern of Os-Fe₂O₃ display a complete reduction process when the temperature reached 700 °C but appearance of weak crystalline FeO observed in the XRD experiment explained the opposite. By addition of the noble metals, the order of the catalyst reducibility is as follows Ru-Fe₂O₃> Os-Fe₂O₃ >Ag-Fe₂O₃.



Fig. 5. TPR profile of (a) Fe_2O_3 , (b) Ru- Fe_2O_3 , (c) Os- Fe_2O_3 and (d) Ag- Fe_2O_3 ,

D. Activation Energy (E_a)

Since Ru doped Fe₂O₃ give complete reduction amongs all

samples, it is appropriate to study the kinetic for further details. According to Wimmers's method [13], the activation energy can be calculated from TPR data by using equation bellow:

$$\ln \left(\Psi/T_{\text{max}}\right) = -E_a/RT_{\text{max}} + \ln \left(AR/E_a\right) + C \qquad (1)$$

The activation energy is achieved from the shift of rate maximum temperature (T_{max}) against heating rate (Ψ) . If a straight line graph is obtained from the plot of ln (Ψ/T_{max}) versus 1/ (T_{max}) , the slop is E_a/R which R is the gas constant. For this study, to evaluate the activation energy of Ru-Fe₂O₃

and undoped Fe₂O₃ a TPR measurement with various heating rate (10, 13 and 15 0 C/min) were carried out. The results showed that by increasing the heating rate, the peaks showing a maximal temperature were shifted to higher temperature as shown in Fig. 6. The activation energy, E_a calculated are accordingly to each steps as below:

$$\operatorname{Fe}_2\operatorname{O}_3 \xrightarrow{\operatorname{K}_{\mathrm{II}}} \operatorname{Fe}_3\operatorname{O}_4 \xrightarrow{\operatorname{K}_{\mathrm{III}}} \operatorname{Fe}\operatorname{O} \xrightarrow{\operatorname{K}_{\mathrm{III}}} \operatorname{Fe}$$

Fig. 7. display the Arrhenius plot of the reduction process which is plotted according to (1) and the activation energies of all reduction steps for undoped Fe_2O_3 and $Ru-Fe_2O_3$ can be calculated form the slop. The E_a of undoped Fe_2O_3 and $Ru-Fe_2O_3$ by referring to their transition phase was summarize in Table II. The results suggested that by adding Ru metal into iron oxide will lower the reduction temperature owing to the decrease of the Ea value by 4.4% to 8.3%. This also in agreement with previous work by Ryu et. al. (2008), they use Rh as a metal additive to lower the reduction temperature of iron oxide in H₂ atmosphere [13].







Fig. 7. Temperature-programmed Arrhenius plots for reducing $Fe_2O_3 \rightarrow Fe_3O_4$ (a) Fe_2O_3 , (b) Ru- Fe_2O_3 , reducing $Fe_3O_4 \rightarrow FeO$ (c) Fe_2O_3 , (d) Ru- Fe_2O_3 , and reducing $FeO \rightarrow Fe$ (e) Fe_2O_3 , (f) Ru- Fe_2O_3 .

TABLE II: THE $E_{\scriptscriptstyle A}$ of Fe_2O_3 and $Ru\mbox{-}Fe_2O_3$ According to the Transition

Sample	$Fe_2O_3 \rightarrow Fe_3O_4$	Fe ₃ O ₄ →FeO	FeO→Fe
	E _a ^I (kJ mol ⁻¹)	$E_a^{II}(kJ mol^{-1})$	E _a ^{III} (kJ mol ⁻¹)
Ru-Fe ₂ O ₃	135	78	92
Fe ₂ O ₃	141.2	82.5	100.3

Furthermore, in Table II, it has been seen that the activation energy for reduction steps of $Fe_2O_3 \rightarrow Fe_3O_4$ is higher compare to the two subsequent reduction steps $Fe_3O_4 \rightarrow FeO \rightarrow Fe$. The values were unpredicted but somehow was similar to the activation energy obtained by [14] for reduction of fresh $Fe_2O_3 \rightarrow Fe_3O_4$ give 139.2 kJ mol⁻¹ and $Fe_3O_4 \rightarrow FeO$ and $FeO \rightarrow Fe$ were 77.3 and 85.7 kJ mol⁻¹, respectively.

IV. CONCLUSION

The Ru-Fe₂O₃, Os-Fe₂O₃, Ag-Fe₂O₃ samples were, respectively, prepared by impregnating the Fe₂O₃ powders with an aqueous solution containing the corresponding metal cations. Based on the results of XRD and TPR, it was found that 3 mol % Ru-Fe₂O₃ gives the best reducibility among all the samples with complete reduction to metallic iron was achieved at lower temperature (650 °C) with only metallic Fe display compare to the undoped Fe₂O₃ (900 °C). While, addition of Os and Ag to the iron oxide inhibit complete reduction to metallic Fe by stabilizing the intermediate FeO. Furthermore, better reducibility of Ru-Fe₂O₃ was also due to the higher in surface area and the decrease of the activation energy regarding to all transition phases.

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