Deactivation Behavior of K$_2$CO$_3$ Catalyst in the Steam Gasification of Kideco Coal

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Abstract >> The present work investigates the effect of K$_2$CO$_3$ catalyst on steam gasification of Kideco coal and the deactivation of the catalyst due to thermal exposure and interaction with coal ash. The gasification reactivity at 700°C is highly enhanced by K$_2$CO$_3$, which is not deactivated by the heat treatment at $T \leq 800$°C. TGA and XRD results prove minor decomposition of K$_2$CO$_3$ after the calcination at 800°C. K$_2$CO$_3$ is, however, evaporated at the higher temperature. Assuming the conversion of K$_2$CO$_3$ into K$_2$O by the decomposition and into K$_2$O·2.5SiO$_2$ and KAlO$_2$ by the interaction with coal ash, the reactivity of the gasification is evaluated in the presence of K$_2$O, K$_2$O·2.5SiO$_2$ and KAlO$_2$. Among them, K$_2$O is the most active, but much lower in the activity than K$_2$CO$_3$. XRD results show that K$_2$CO$_3$ could react readily with the ash above 700°C.

Key words: Steam gasification(스팀가스화), Catalyst(촉매), Coal(석탄), Deactivation(비활성화), Potassium(칼륨)

1. INTRODUCTION

Coal is one of the most important energy sources, currently accounting for ~70% of proven fossil fuel resources and also ~25% of worldwide energy consumption$^{1-3}$. In the medium to long-term aspect, coal is expected to remain a main energy source for the traditional industries such as electricity generation, iron smelting, and cement manufacturing, thanks to its widespread abundance and economic benefit.

Gasification of coal has gained popularity, as it is more energy-efficient and can reduce the GHGs emission$^{4,7}$. Many of the commercial gasification processes are being operated at $T > 1200$°C in order to boost gasification rate$^{8-10}$. This severity requires exothermic combustion of coal and therefore lowers the overall efficiency. The conversion kinetics at the lower
temperature \((T < 900^\circ C)\) can be improved by introduction of catalytically active potassium \(^{11-13}\). Potassium catalyst is fed to the reactor, typically in the form of potassium carbonate (\(K_2CO_3\)) \(^{14}\). However, the repeated use of \(K_2CO_3\) seems to be difficult, mainly due to deactivation of the catalyst by both thermal degradation and irreversible interaction with the mineral matters in coal \(^{15-17}\). In the process of the catalytic coal gasification, \(K_2CO_3\) can be decomposed into \(K_2O\). Moreover, \(K_2O\) can react with coal ash that is composed of mostly \(Al_2O_3\) and \(SiO_2\), forming minerals such as \(K_2O\cdot xSiO_2\), \(KAlO_2\), Sanidine (\(K_2O\cdot Al_2O_3\cdot 6SiO_2\)), Osumilite (\(K_2O\cdot 4.4MgO\cdot 4.6Al_2O_3\cdot 20.4SiO_2\)), and etc. The catalytic behavior of these potassium compounds is not well known.

In this work, the catalytic activity of \(K_2CO_3\) calcined at \(800^\circ C\), compared to that of fresh \(K_2CO_3\), is evaluated and discussed based on the results from TGA, SEM, and XRD. In addition, the activity of various deactivated forms of \(K_2CO_3\) (\(K_2O\), \(K_2O\cdot 2.5SiO_2\), and \(KAlO_2\)) is tested at \(700^\circ C\), feeding steam, and compared with non-catalytic case. After 5 hr steam gasification of Kideco raw coal in a fluidized bed (FB) reactor, the used \(K_2CO_3\) catalyst, and the unreacted coals residing in the reactor and the back filter were collected \(^{18}\). The chemical composition of the used \(K_2CO_3\) as well as the reactivity of the unreacted coals were then evaluated.

### 2. EXPERIMENTAL

#### 2.1 Materials

Kideco coal was used as a gasification sample. It is low rank coal, and its proximate/ultimate analysis and calorific value are tabulated in Table 1. \(K_2CO_3\) (Showa Chemical Inc.), \(K_2O\) (96.5% purity, Alfa Aesar), \(K_2O\cdot 2.5SiO_2\) (Alfa Aesar) and \(KAlO_2\) (high purity, Kojundo Chemical Laboratory Co.) were chosen as potassium source for the catalytic reaction. Thermally stressed \(K_2CO_3\) was prepared by heat treatment for 3 hr at four different temperatures (600, 700, 800, and \(900^\circ C\)) with 0.36 mL/hr steam flow in 100 cc/min \(N_2\).

Coal ash was obtained from combustion of Posco coal and its composition is given in Table 1 (proximate/ultimate analysis and calorific value) and Table 2 (chemical composition of Posco coal ash). Two different locations in a SEM image in Fig. 1 were chosen for the composition analysis using EDS. The main components are \(SiO_2\) and \(Al_2O_3\). A small quantity of other metals, such as K, Fe, Na, Ti, and Mg is also included.

#### 2.2 Steam Gasification of Coals

Steam gasification of Kideco coal was performed

### Table 1 Proximate/ultimate analysis and calorific value of Kideco raw coal

| sample  | Proximate analysis (wt.%) | Ultimate analysis (wt.%) daf | CV (kcal/kg) |
|---------|--------------------------|-----------------------------|--------------|
| M VM*   | Ash*                     | FC*                         | C H N O S    |
| Kideco  | 27.2 45.9 6.3 47.7 70.3 5.1 1.2 23.2 0.2 | CV | 4,410 |
| Posco   | 1.3 21.3 9.8 68.9 86.2 4.1 1.5 1.1 7.1 | 8,596 |

(M: moisture, VM: volatile matter, FC: fixed carbon, *: dry basis, daf: dry & ash-free, CV: calorific value)
Table 2 Chemical composition of ash from Posco coal

| wt.%  | SiO₂ | Al₂O₃ | K₂O | Fe₂O₃ | Na₂O | TiO₂ | MgO |
|-------|------|-------|-----|-------|------|------|-----|
| A-site| 83.3 | 10.6  | 2.5 | 1.9   | 1.6  | -    | -   |
| B-site| 63.5 | 27.0  | 3.7 | 3.4   | 0.5  | 0.6  | 1.4 |

in a fixed bed reactor\(^7\). A coal was pulverized solely or together with a catalyst and meshed to < 75 μm. The sample (0.1 g) was introduced into a quartz reactor. The gasification was carried out at 700°C with 30 vol.% steam in 100 cc/min N\(_2\). Temperature was increased to the target at a ramp rate of 30°C/min. Produced gases (H\(_2\), CH\(_4\), CO, and CO\(_2\)) were quantified every 12 min using a gas chromatography (GC, Agilent 6890). GC used for this experiment has two separation columns: a front column was porapak N to quantify H\(_2\) and CH\(_4\) using N\(_2\) reference gas, and a back column was porapak Q in series with molsieve to quantify CO and CO\(_2\) using helium reference, where thermal conductivity detectors (TCD) were adapted for both columns. The oven temperature was set to 35°C and the sampling interval of 12 min was used. Water was supplied to the reactor by a syringe pump, which was vaporized in heated stainless steel tubing (150°C) and delivered to the reactor by 100 cc/min N\(_2\). Side products, such as water and tar, were removed by 2-step cold traps (2°C) and an oil filter. Only gaseous products were transferred to the GC. GC was calibrated with standard gas 0.1% and 3.0% containing H\(_2\), CH\(_4\), CO and CO\(_2\) with N\(_2\) carrier. The conversion percentage was calculated as the accumulated amount of the product gases divided by the total amount of the gases produced.

2.3 Characterization

Proximate analysis was done by TGA-701 Thermogravimeter (LECO Co., USA) base on ASTM D3172, while ultimate analysis was done by using CHN-2000 Elemental Analyzer (LECO Co., USA). Calorific value was determined using Parr 1261 Calorimeter (PARR Co., USA). Thermogravimetric analysis (TGA) of K\(_2\)CO\(_3\) was performed under air, N\(_2\), and CO\(_2\) atmosphere using SDT 2960 simultaneous DTA-TGA (TA instruments). About 10 mg sample was loaded and temperature was increased to 900°C at 10°C/min ramp rate. SEM image of fresh and 800°C calcined K\(_2\)CO\(_3\) was taken using Hitachi S-4800 machine. XRD pattern was obtained using Rigaku Dmax-2500 for K\(_2\)CO\(_3\) calcined at 600, 700, and 800°C, and a mixture of K\(_2\)CO\(_3\) and coal ash calcined at 700, 800, and 900°C.

3. RESULTS AND DISCUSSION

3.1 Thermally Treated K\(_2\)CO\(_3\) Catalyst

The conversion profile of steam gasification at 700°C is provided for Kideco raw coal without, and with fresh and 800°C-calcined K\(_2\)CO\(_3\) catalyst (5.7 wt.%, metal basis) (Fig. 2).

Potassium carbonate was chosen as a catalyst, since
it is one of the most pronounced catalysts for coal gasification and is free from the mass transport limitation\(^{13}\). The calcination temperature (800°C) was set to be higher than the reaction temperature (700°C) in order to see the sole effect of thermal stress on the catalytic activity of K\(_2\)CO\(_3\). The reaction catalyzed by fresh K\(_2\)CO\(_3\) shows much faster kinetics than the non-catalytic one. This happens via the catalytic dissociation of the oxygen-containing molecules, which provides a lower energy pathway and thereby increases the oxygen concentration on the carbon surface through an oxidized catalyst-carbon intermediate\(^{19}\). The deactivation is not found by calcination of K\(_2\)CO\(_3\) at 800°C. The conversion reaches > 90% after 60 min similarly for both fresh and the thermally stressed K\(_2\)CO\(_3\). On the other hand, without the catalyst, ~40% of Kideco coal is gasified after 60 min and ~72% after 156 min. Further analysis on the thermal behavior of K\(_2\)CO\(_3\) was done using TGA, SEM and XRD.

TGA analysis was done to determine the behavior of K\(_2\)CO\(_3\) at the elevated temperature under air, N\(_2\) and CO\(_2\) atmosphere. Fig. 3 shows thermal variation of weight of K\(_2\)CO\(_3\).

In general, similar TGA profiles are shown between the different atmospheres; a steep drop at T < 200°C followed by a flat region at T = 200~800°C and a mild fall at T > 800°C. A rapid loss at T < 150°C for air and N\(_2\) is most likely due to evaporation of adsorbed water, and which does not appear after 200°C. A peculiar increase of the weight is shown up to 100°C under CO\(_2\), seemingly indicating the carbonate formation or physi-sorption of CO\(_2\)\(^{20}\). It loses weight from 100 to 160°C like under air and N\(_2\). An absolute quantity of the loss under CO\(_2\) is ~2% less than under air and N\(_2\). In the temperature range of 200~800°C, no significant change is found for all the cases. Further weight loss happens above 800°C possibly because of the decomposition of K\(_2\)CO\(_3\) by reaction (1) and the evaporation near a melting point (891°C\(^{21}\)).

\[
\text{K}_2\text{CO}_3 \rightarrow \text{K}_2\text{O} + \text{CO}_2
\] (1)

Based on the TGA results, K\(_2\)CO\(_3\) is stable against thermal treatment at 800°C, in terms of the chemical composition. Therefore, catalytic gasification using K\(_2\)CO\(_3\) can be performed below 800°C without worrying about the degradation due to the chemical decomposition.

The effect of thermal treatment on microstructure of K\(_2\)CO\(_3\) was studied by looking at SEM images of fresh and 800°C-calcined K\(_2\)CO\(_3\) (Fig. 4).

Compared to amorphous and continuous surface of fresh K\(_2\)CO\(_3\), the heat-treated one contains well-defined grains (< 1 µm) with pores that might work as a pathway of gaseous participants. Cracks are found at the surface of fresh K\(_2\)CO\(_3\). The microstructural effect on the reactivity seems to be minor in this system, since the solid-solid contact of Kideco coal and K\(_2\)CO\(_3\) happens with larger size of the particles (10~75 µm) rather than microstructural grains. Moreover, consid-
eration of the microstructure becomes insignificant in case of the gasification in a fluidized bed reactor. Because a bead-type catalyst sized > 1 mm would contact with coal reactants.

XRD patterns of heat-treated pure \( \text{K}_2\text{CO}_3 \) were obtained (Fig. 5). It was calcined at 600, 700, 800, and 900°C. After calcination at \( T = 600 \) and 700°C, the peaks corresponding to \( \text{K}_2\text{CO}_3 \) and \( \text{K}_2\text{CO}_3\cdot1.5\text{H}_2\text{O} \) appear and the two temperatures exhibit about the same profile. \( \text{K}_2\text{CO}_3 \) calcined at 800°C shows the peaks mainly due to \( \text{K}_2\text{CO}_3\cdot1.5\text{H}_2\text{O} \) with much weaker \( \text{K}_2\text{CO}_3 \) and there is no peaks indicative of the decomposition (reaction (1)).

When heat-treated at 900°C under steam/N\(_2\) atmosphere, solid \( \text{K}_2\text{CO}_3 \) was completely evaporated and all gone\(^{22}\). Considering the melting point of \( \text{K}_2\text{CO}_3 \) (891°C), it seems to be safe that the catalytic gasification using \( \text{K}_2\text{CO}_3 \) is performed below 850°C, since the volatility of \( \text{K}_2\text{CO}_3 \) varies depending on the atmospheric condition\(^{21}\). This result points out that a weight loss shown in TGA profile at \( T > 800^\circ\text{C} \) (Fig. 3) might be partly due to the evaporation along with the decomposition (reaction (1)).

### 3.2 Deactivation of \( \text{K}_2\text{CO}_3 \) by Irreversible Interaction with Coal Ash

\( \text{K}_2\text{CO}_3 \) has been known to catalyze the coal gasification via its redox cycle, being reduced by carbon and oxidized by steam\(^{23}\). However, it would be easily deactivated\(^{15,24}\). Either the decomposition or mineralization happens, forming new potassium compounds probably containing Si and Al, since the major components in coal ash are silica and alumina. The minerals produced are typically complicated, sometimes combined with other metallic species; for example, Osumilite (\( \text{K}_2\text{O}\cdot4.4\text{MgO}\cdot4.6\text{Al}_2\text{O}_3\cdot20\text{SiO}_2 \)), Sanidine (\( \text{K}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot6\text{SiO}_2 \)), and Nepheine (\( \text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot2\text{SiO}_2 \))\(^{15}\). In this work, simplified products of the deactivation of \( \text{K}_2\text{CO}_3 \), such as \( \text{K}_2\text{O}, \text{K}_2\text{O}\cdot2.5\text{SiO}_2 \), and \( \text{KAIO}_2 \), are evaluated as a source of potassium at 700°C (Fig. 6).

Potassium content (metal basis) is 4.6~7.1 wt.%; 5.7 wt.% for \( \text{K}_2\text{CO}_3 \) and \( \text{K}_2\text{O} \), 4.6 wt.% for \( \text{K}_2\text{O}\cdot2.5\text{SiO}_2 \), and 7.1 wt.% for \( \text{KAIO}_2 \). As expected, \( \text{K}_2\text{CO}_3 \) shows the best performance with > 90% conversion within 60 min. Both \( \text{K}_2\text{O} \) and \( \text{K}_2\text{O}\cdot2.5\text{SiO}_2 \) give ~45% conversion after 60 min. Since then, \( \text{K}_2\text{O} \) exhibits higher conversion (> 90% at 132 min) than \( \text{K}_2\text{O}\cdot2.5\text{SiO}_2 \) (~76% at 132 min). On the other hand, \( \text{KAIO}_2 \) is almost non-catalytic, showing a conversion profile similar to Kideco coal.
without a catalyst. The deactivation of K$_2$CO$_3$ via the decomposition to K$_2$O and the K$_2$CO$_3$–ash reactions observed in the work agrees with the previous works\(^{125}\). In reality, even more severe and broader deactivation due to the reactions with various ash components are expected, limiting repeated use of K$_2$CO$_3$. It seems to be very difficult to overcome the deactivation if ash-containing coals are gasified. Use of ash-free coal substrates could be a solution for the issue\(^{7}\).

Ash from Posco coal was mixed with K$_2$CO$_3$ and the mixture (1:1 weight ratio) was calcined at 600, 700, 800, and 900°C for 3 hr under 30 vol.% steam in N$_2$. In order to investigate the K$_2$CO$_3$–ash reactions, XRD of the samples was taken, as shown in Fig. 7. No significant reactions are observed from the mixture at 600°C, such that the peaks arise from hydrous K$_2$CO$_3$ and silica. XRD profile at T $\geq$ 700°C is different from at 600°C. The peaks show the new phases, especially at $2\theta$ = 29.5° and 34.2° and reduced intensity of K$_2$CO$_3$ and silica is also shown. The general profile at T = 700~900°C is about the same with each other, showing that the K$_2$CO$_3$–ash reactions start from 700°C.

### 3.3 Used K$_2$CO$_3$ Catalyst at 5 hr Gasification in a Fluidized-bed Reactor

Steam gasification of Kideco coal catalyzed by K$_2$CO$_3$ was done for 5 hr in a fluidized bed (FB) reactor under the following condition; 800°C, 4 atm, coal feed rate = 1 kg/hr, oxygen to carbon ratio (O$_2$/C) = 43%, and steam to carbon ratio (H$_2$O/C) = 108.7%\(^{18}\). At the completion of the reaction, used K$_2$CO$_3$ catalyst was collected from the FB reactor and its chemical composition was analyzed using XRD (Fig. 8). It shows similar profile with K$_2$CO$_3$ calcined at 800°C and most of

![Fig. 7 XRD pattern of a mixture of K$_2$CO$_3$ and ash heat-treated at 600, 700, 800 and 900°C](image-url)

![Fig. 8 XRD pattern of used K$_2$CO$_3$ after 5 hr reaction in a fluidized bed at 800°C](image-url)
the peaks correspond to $\text{K}_2\text{CO}_3$ and silica. The new phases as a result of $\text{K}_2\text{CO}_3$-ash reactions, such as the peaks at $2\theta = 29.5^\circ$ and $34.2^\circ$ in Fig. 7, are not detected even after the 5 hr reaction. Insufficient contact between the catalytic bead and the coal seems to minimize the irreversible reaction and thus the deactivation.

Unreacted Kideco coal both in a FB reactor (namely Char) and a metal filter (namely Filter) were also recovered. When the samples are gasified in a fixed bed reactor, much reduced reactivity, compared to the raw coal, appears for both of the coals (Char and Filter), as shown in Fig. 9. “Char” is gasified so slowly that the conversion is only ~8% at 700°C, ~13% at 750°C, and ~40% at 800°C after 156 min. “Filter” is a little faster than “Char”: ~30% at 700°C, ~59% at 750°C, and ~92% at 800°C. Both of them are much slower than Kideco raw coal that shows > 70% conversion at 700°C after 156 min (Fig. 2). Obviously, a reactive portion of the coal is gasified earlier in the FB process and the remaining “Char” and “Filter” become less reactive.

4. CONCLUSION

Commercial catalytic gasification of coal has not been maximally achieved because of thermal and chemical deactivation of the catalyst. In the work, stability of $\text{K}_2\text{CO}_3$, one of the most popular catalysts, is evaluated. The enhanced kinetics is observed for fresh $\text{K}_2\text{CO}_3$, which maintains the activity even after the calcination at 800°C. TGA and XRD results confirm that no decomposition of $\text{K}_2\text{CO}_3$ happens below 800°C. However, $\text{K}_2\text{CO}_3$ should be used probably below 850°C, due to the loss of the catalyst by evaporation at the higher temperature. The reactivity is not decreased by the grain growth of $\text{K}_2\text{CO}_3$. XRD results identify the reaction of $\text{K}_2\text{CO}_3$ with coal ash above 700°C, deactivating the catalyst. The activity of potassium varies according to its chemical forms and it decreases in the order: $\text{K}_2\text{CO}_3 > \text{K}_2\text{O} > \text{K}_2\text{O} \cdot 2.5\text{SiO}_2 > \text{KAIO}_2$ where $\text{KAIO}_2$ is almost non-catalytic. Repeated use of $\text{K}_2\text{CO}_3$ catalytic bead might be possible when gasified at < 800°C in a fluidized bed.

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