Promotional effect of Au on γ-Al₂O₃ supported cobalt based catalyst for total oxidation of methane

M. Kumar[1,2*], G. Rattan[1], R. Prasad[3]

[1]*Dr. S. S. Bhatnagar University Institute of Chemical Engineering & Technology, Panjab University, Chandigarh 160014, India*
[2]*Department of Chemical Engineering, Chandigarh University, Mohali, Punjab, India*
[3]*Department of Chemical Engineering, IIT-BHU, Varanasi, U.P, India*

Effect of gold loading (within the range of 4 mass % - 16 mass %) on the behavior of the catalyst (Co/γ-Al₂O₃) for CH_4 oxidation is studied. Gold promoted CoO_x/γ-Al₂O₃ catalysts were prepared *via* deposition precipitation method and calcined at 550ºC. A significant improvement in activity was observed ($T_{100\%}$=380 ºC) toward CH_4 oxidation with increase in gold loading, though a plateau was reached at ca. 13.45 wt. % Au content. The catalytic activity is studied on a fixed catalyst weight (500 mg) in an air flow containing 1 % of CH_4 introduced to the reactor at a total feed rate of 150 mL/min. The catalyst characterization is done using XRD, TGA/DSC and SEM. The best catalysts according to the catalytic activity obtained are as follows: Au (13.45>10.44>16.27>07.21>04.06>0 wt. %)-CoO_x/γ-Al₂O₃. It is found that the catalyst containing 13.45 wt. % of Au loaded on 18 wt. % Co/γ-Al₂O₃ is the most active catalyst as it decreases the temperature ($T_{100\%}$=380 ºC) by 110ºC required for methane oxidation in comparison to the catalyst that does not contain gold, i.e., 18wt.%Co/γ-Al₂O₃ ($T_{100\%}$=490 ºC).

Keywords: Methane, Oxidation, Catalysts, Gold, Cobalt, Alumina

INTRODUCTION

With the exponential growth of vehicles on road, fossil fuels have led to an alarming level of exploitation. Thus the need arises for substitution of both, the vehicles and the fuel in pursuit of green and clean environment. In comparison to gasoline and diesel, compressed natural gas (CNG) as a fuel is an attractive choice with significant environmental advantages as it is available in abundance, is cheaper and highly efficient.
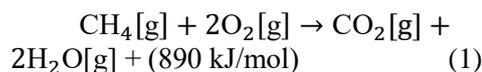
According to Jahirul *et al*. [1] it suitably mixes with air and burns effectively, due to its high octane number, engine operates smoothly without knocking, the formation of non-methane hydrocarbons and other air pollutants in CNG fueled engines is one-tenth of that in gasoline engines. The formation of CO and NOx is by 80% lower than in gasoline engines because of the simple chemical structure of compressed natural gas (primarily CH_4), it contains single carbon in comparison to diesel ($C_{15}H_{32}$) and gasoline (C_8H_{18}). In terms of economy CNG fueled engines are by 20% more economical than gasoline and diesel engines [2].

However, the inevitable methane emission from vehicles using CNG as the fuel has a very harmful effect on the environment, as methane is a potent greenhouse gas and its global warming potential is 21 times that of CO_2 at equivalent emissions [3, 27].

Further, compared to low-emitting diesel, the CNG exhaust has higher levels of six toxic air contaminants (TAC) listed by the California Air Resources Board (ARB), namely: acetaldehyde, acrolein, benzene, formaldehyde, methyl ethyl ketone, and propionaldehyde. Formaldehyde emission is very high with CNG vehicles. It is also classified as a toxic air contaminant and is known as carcinogenic [4].

Therefore, the challenge before the researchers around the globe is to abate harmful chemicals from CNG exhaust, especially methane emission. This task can be achieved with the help of a catalytic convertor [5] fitted before the exhaust of vehicle. The convertor has the properties of reducing harmful chemicals to less harmful chemicals (CO_2, H_2O). The oxidation and reduction reactions take place due to the catalyst present on the interior of the surface of convertor. Catalyst present in the interior oxidizes the unburnt methane from vehicular exhaust and converts it into less harmful products (Eq. 1).

$$CH_4[g] + 2O_2[g] \rightarrow CO_2[g] + 2H_2O[g] + (890 \text{ kJ/mol}) \qquad (1)$$

Gold based catalysts have been studied by numerous researchers for oxidation of methane. Numerous PhD degrees have been awarded [6], various patents have been granted [7-8], a number of research [9-16] and review [17-19] articles has been published on gold based catalysts for methane

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* To whom all correspondence should be sent:
E-mail: maninderbhatoy@gmail.com

oxidation/combustion reactions.

Table 1 depicts some of the advances made on gold based catalysts and is strictly devoted to gold based catalysts. A number of conclusions can be drawn from this table such as preparation methods of the catalyst, type of the reactor used, amount of catalyst used for reaction, flow conditions and at last but not least the T$_{100\%}$ which denotes the temperature required for 100% conversion of methane. It is interesting to note that from all the reported work, the minimum temperature for 100% conversion of CH$_4$ is 350 °C [12] and the maximum hovers around 680 °C [20]. However, all the other works report an average temperature of 500 °C. Keeping in view the literature accumulated it was observed that the combination of gold, cobalt and γ-alumina has not been investigated over the whole composition range. Hence, the present work is devoted to the promotional effect of gold on alumina supported cobalt based catalyst.

EXPERIMENTAL

Catalyst preparation

The Au-Co/γ-Al$_2$O$_3$ mixed oxide catalysts were prepared by a deposition precipitation method [5]. All chemicals used were of analytical grade (AR). Stoichiometric amount of gold (HAuCl$_4$) and cobalt (Co(NO$_3$)$_2$.6H$_2$O) salts were first dissolved in doubly distilled water followed by addition of γ-alumina powder. The formed suspension was stirred vigorously at 70°C. Sodium carbonate solution was added dropwise to this suspension maintaining pH of 7, hence the formation of precipitates takes place. The precipitate was washed several times with deionized water. The formed solid was heated overnight in an oven at 110 °C. Further it was pressed (mortar and pestle) and finally the fine powder was calcined at 550 °C for 4 h.

Table 1. Parameters in some publications related to gold based catalysts

Catalyst, Prepn. Method	Exp. Operating Parameters	Remark	Reference
Au/Co$_3$O$_4$–CeO$_2$ Co-precipitation, Calc. temp. 600 °C.	50 mg, U-shaped quartz, 0.3 vol.%CH$_4$, 2.4 vol.%O$_2$ in He. WHSV=60,000 mL/ gh.	AuCo$_3$O$_4$ T$_{100\%}$=550°C.	[11]
Au/CoOx, Au/MnO$_x$ Co-precipitation, Calc. temp. 400 °C	50 mg, fixed-bed S. S. tubular flow, 50 mL/min, GHSV = 15,000 h^{-1}, alkane/air (molar ratio) of 0.5/99.5	Au/CoO$_x$ T$_{100\%}$= 350 °C.	[12]
Au/MgO, Impregnation, Calc. temp. 850 °C.	200 mg, microreactor of quartz glass, CH$_4$ (46%, O$_2$ 8%, He 46%. GHSV=750 h^{-1}.	Enhanced activity when gold was employed	[21]
Au–Pt/Co$_3$O$_4$ Co-precipitation technique Calc. temp. 400 °C.	0.10 g, fixed-bed microreactor, 1 vol.% CH$_4$, 5 vol.% O$_2$, and rest N$_2$, space velocity of 10,000 h^{-1}	Activity of Au/Co$_3$O$_4$ enhanced when Pt, e.g. 0.2 wt.% was added, and conv. temp. decreased by 50°C.	[22]
Au/MOx/Al$_2$O$_3$ where M is Cr, Mn, Fe, Co, Ni, Cu, and Zn, Homogeneous deposition precipitation, Calc. temp. 400 °C	Lab-scale fixed-bed, 0.8 vol.% CH$_4$ and 3.2 vol.% O$_2$ in He, 30 mL/min.	Au/Al$_2$O$_3$ T$_{100\%}$= 655°C	[23]
Au/TiO$_2$, Au/SiO$_2$, Au/CeO$_2$, Au/ZrO$_2$, Au/Al$_2$O$_3$, Deposition precipitation, Calc. temp. 400 °C	Quartz tubular fixed bed, 50 mL/min. (20% CH$_4$ and 5% O$_2$, rest is He).	Au/TiO$_2$ T$_{80\%}$= 400°C, Au/Al$_2$O$_3$ T$_{20\%}$=400°C.	[10]
Au/Fe$_2$O$_3$ Deposition precipitation	0.1 g, fixed-bed quartz, 1 vol.% CH$_4$ in air, flow rate=100 mL/min, GHSV = 51,000 h^{-1}.	Au/Fe$_2$O$_3$ prep. by HDP T$_{50\%}$=375°C, T$_{100\%}$=500°C.	[24]
AuOx/Ce$_{0.6}$Zr$_{0.3}$Y$_{0.1}$O$_2$ Co-precipitation, Calc. temp. 700 °C	0.12 mg, quartz fixed-bed microreactor, 5% CH$_4$/95% He (v/v, 40 mL/min) + O$_2$ (8 mL/min) + He (52 mL/min), SV= 50,000 h^{-1}.	Catalyst with 6% AuO$_x$ T$_{50\%}$=590°C T$_{100\%}$=680°C	[20]

Table 2. Composition of gold on different catalysts with catalyst ID.

Catalyst ID	G50	G51	G52	G53	G54	G55
Gold (wt.%)	00.00	04.06	07.21	10.44	13.45	16.27
Co:Au	5:0	5:1	5:2	5:3	5:4	5:5

Au-CoO$_x$/γ-Al$_2$O$_3$ samples contained {00.00, 04.06, 07.21, 10.44, 13.45, 16.27 wt. % Au} and {18 wt.% Co}. The compositions of gold and cobalt are expressed in weight percent throughout the paper and are also presented in Table 2, each catalyst having a unique ID. The resulting powder was finally stored in air-tight triplex vials for further experiments and characterization.

Catalytic Activity Measurement

The catalytic activities of all prepared catalysts (Table 2) towards methane oxidation were tested in a compact scale fixed-bed, down-flow tubular reactor [5]. The reactor was placed vertically in a split open furnace. 500 mg of the catalyst was diluted with 1 ml of alumina and placed in the reactor. The reactant gas mixture consisted of 1% CH$_4$ in air maintaining a total flow rate of 150 mL/min, corresponding to a WHSV about 36000 mLh^{-1}g^{-1}. The reaction was carried out in temperature range of ambient to a temperature at which 100% CH$_4$ conversion was attained. It was also ensured that the gas does not contain any moisture or CO$_2$ by passing it through CaO and KOH pellet drying towers. Digital flow meters were used for measuring the flow rate of both methane and air. The catalytic experiments were carried out under steady-state conditions. The steady-state temperature was controlled with the help of a microprocessor-based temperature controller with precision of ±0.5℃. The products and reactants were analyzed by means of an online gas chromatograph (Nucon 5765) using a Porapak Q-column, methaniser and FID detector for the concentrations of CH$_4$ and CO$_2$. The fractional conversion of methane was calculated on the basis of the values of concentration of CH$_4$ in the product stream using Eq. (2). Only one chromatogram peaks corresponding to CH$_4$ was recorded for inlet, whereas the chromatogram of reactor outlet gases contains an additional peak of CO$_2$ with unreacted CH$_4$.

$$X_{CH_4} = \left\{ \left| C_{CH_4} \right|_{in} - \left| C_{CH_4} \right|_{out} \right\}_i / \left| C_{CH_4} \right|_{in} \quad (2)$$

where the change in concentration of CH$_4$ due to oxidation at any instant $\left\{ \left| C_{CH_4} \right|_{in} - \left| C_{CH_4} \right|_{out} \right\}_i$ is proportional to the decrease in the area of the chromatogram of CH$_4$ at that instant. Experimental

data were collected at every measurement point after stabilizing for 20 min.

Catalyst Characterization

Characterization of the catalysts was done by XRD (X-Ray Diffraction), TGA/DSC (thermo gravimetric analysis) and SEM (scanning electron microscopy). XRD pattern was recorded in an X'PERT PRO diffractometer using CuKα radiation source. Operating current and operation voltage were 40mA and 45KV, respectively. The scanning range 2Ө was 5.00 - 99.98º with a divergence slit of 0.8709º. The continuous scanning was done with a step size of 0.0170º and with scan step time of 30.36 s. The TGA/DSC analysis was carried out in air on a thermo gravimetric analyzer. The temperature of the cycle was programmed from 40 to 700˚C increasing at the rate of 10˚C per minute. In order to observe the microstructure of the prepared catalysts, SEM micrographs were obtained using a JEOL JSM-6700F instrument.

RESULTS & DISCUSSION

Effect of gold addition to CoO$_x$/γ-Al$_2$O$_3$

In Figure 1, the methane conversion is plotted as a function of the operating temperature for each catalyst. It was observed that the methane conversion increases by increasing temperature. This evidence can be explained by considering that with increase in temperature, the activation energy required for the reaction is attained in that temperature region. It can be seen that with increase in gold content, the activity of the catalyst increases. The plot (figure 1) can be divided into two branches: methane conversion below 50% and methane conversion above 50%. It can be seen that below 50% the ignition temperature for methane oxidation is almost the same for each of the catalysts. But with increase in temperature each curve proceeds *via* different oxidation route.

Reaching 50% methane conversion, each curve differs by 25-30 ºC, as far as methane conversion above 50% is concerned. The difference of conversion temperature is minimised with increase in temperature. Each catalyst has the ability for 100% methane conversion. Further, it can be seen that almost each catalyst has traced an S-type curve. It is observed that the catalyst having 13.45% Au loaded on 18 wt.% CoO$_x$/γ-Al$_2$O$_3$ exhibits the highest activity among all prepared catalysts. The

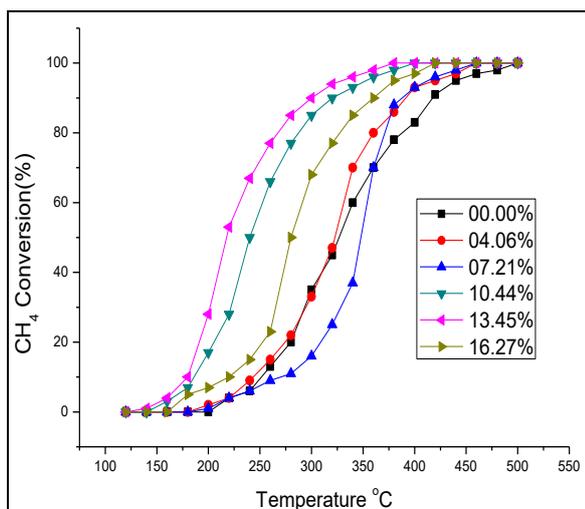catalyst without gold has the worst activity among all catalysts.

Figure 1. Temperature dependence of methane conversion over different amounts of gold loaded on 18wt.% Co/γ-Al₂O₃.

A significant influence of the gold loading on the activity of different catalysts is evident. It is interesting to note that the catalyst (G50) without gold shows high activity than the catalyst (G52) between the span of 260-340°C temperature. It is very clear from figure 2 that with increase in gold content the activity of catalyst increases up to 13.45%, however, on further increase in gold content the activity decreases. This can be attributed to the bulk dispersion or aggregated particles of gold with further increase in gold content. The activity of the highly active catalyst (G54) can be attributed to the uniform dispersion of gold and cobalt on γ-alumina.

X-Ray diffractograms (XRD)

The XRD patterns of Au-CoOₓ/γ-Al₂O₃, as well as of CoOₓ/γ-Al₂O₃ are shown in figure 3 and figure 4, respectively. The formation of crystalline phase can be seen from the patterns of powders calcined at 550°C. The peaks at 2θ=45. 65° and 67.18 (JCPDS 04-0783) are attributed to alumina in all catalysts [25,6].

The intense peaks further correspond to 38.2° the (111) lattice planes of metallic cobalt phase [26]. Narrow peaks indicate large particles and broad peaks correspond to small particles.

Characterization of the pure Co₃O₄/γ-Al₂O₃ catalysts in comparison with catalyst having Au loadings by powder X-ray diffraction showed that Au phases could be observed, although the Au loading in the catalyst (G55) was 16.27 wt.%, and only Co₃O₄/γ-Al₂O₃ was determined, indicating that Au was amorphous or highly dispersed.
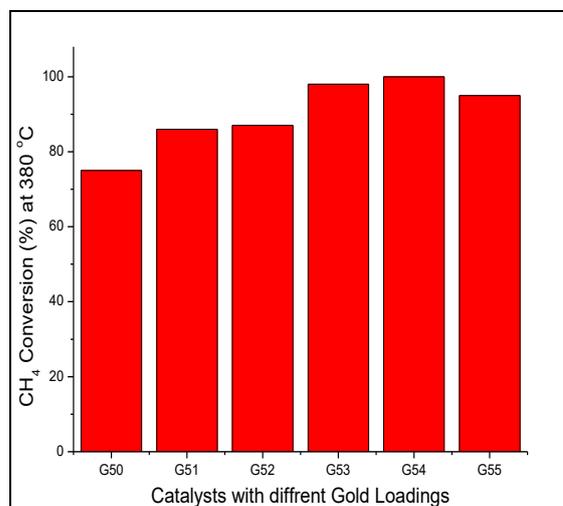

Figure 2. Methane conversion at 380°C for all catalysts.

Thermo Gravimetric Analysis (TGA/DSC)

The result of the TGA/DSC analysis of G53 catalyst is shown in figure 5. There is approx. 12wt.% reduction in weight of the catalyst. It can be seen that weight loss occurs in three intervals, i.e. 40-270; 270-550; 550-690 °C.

From 40-270 °C the weight loss is significant and may be attributed to the loss of water present with the precursor with an endothermic peak at 270°C. With further increase in the temperature there is a tremendous decrease in weight loss in the temperature range of 270-550 °C. This is due to the decomposition of nitrates and chlorides present in the precursor on contact with oxygen. However, the weight loss between 550-690 °C is negligible which means that anhydrous salts are completely decomposed. Figure 6 represents the thermograms of G54 catalyst. The weight loss is around 12 wt%, a little more than G53. This may be due to the high concentration of gold. The weight loss trend is similar to G53 catalyst. However, much of the weight loss is in the range of 40-270 °C with the endothermic peak again at 270°C. After 550 °C the loss in weight is negligible.
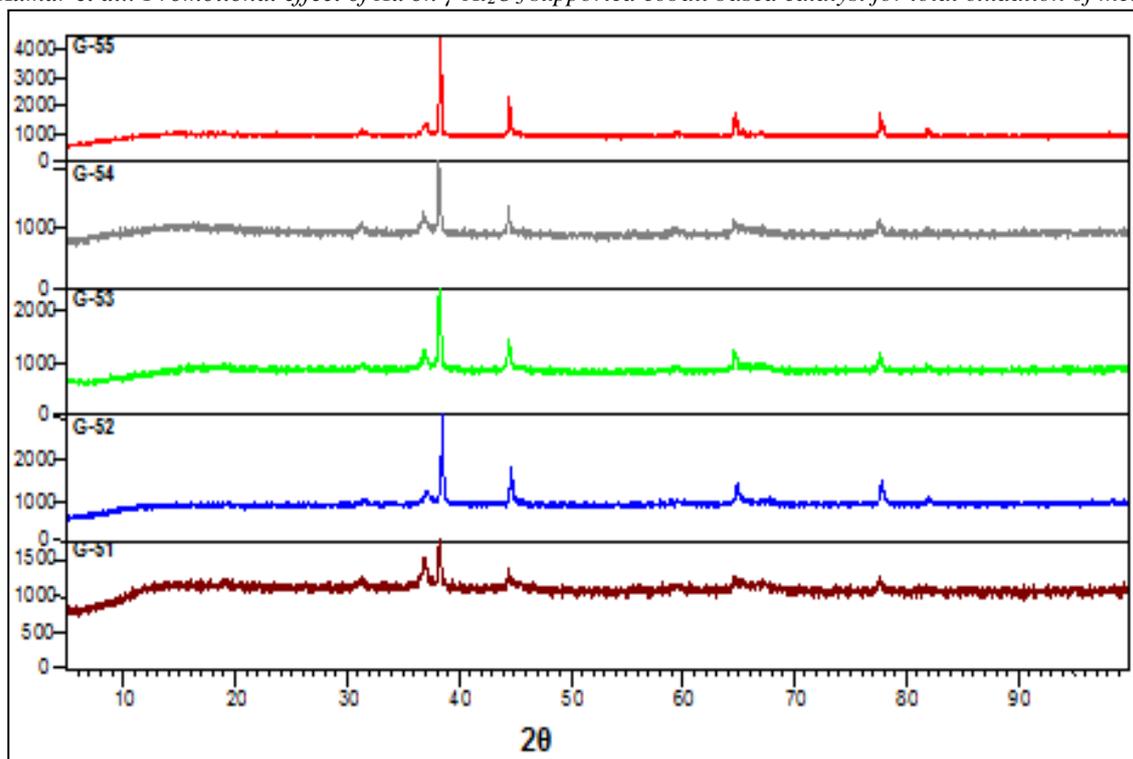
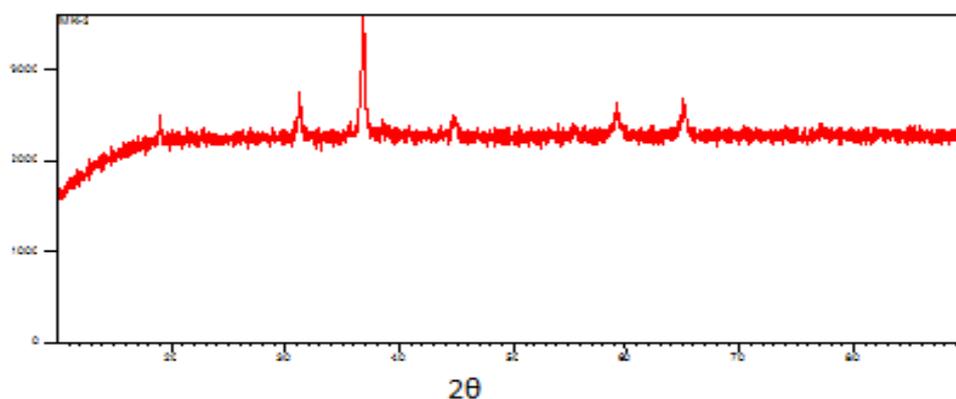**Figure 3.** XRD patterns of catalysts having different gold loadings**.**

Figure 4. XRD pattern of 18wt.% Co/γ-Al₂O₃

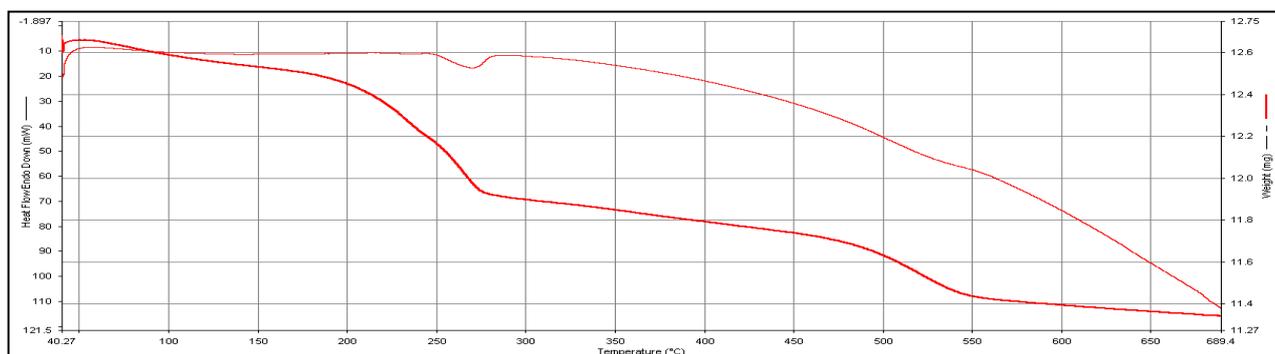**Figure 5.** Graphical representation of TGA/DSC thermograms of the precursors of 10.44wt.% Au supported on 18wt.%Co/γ-Al₂O₃
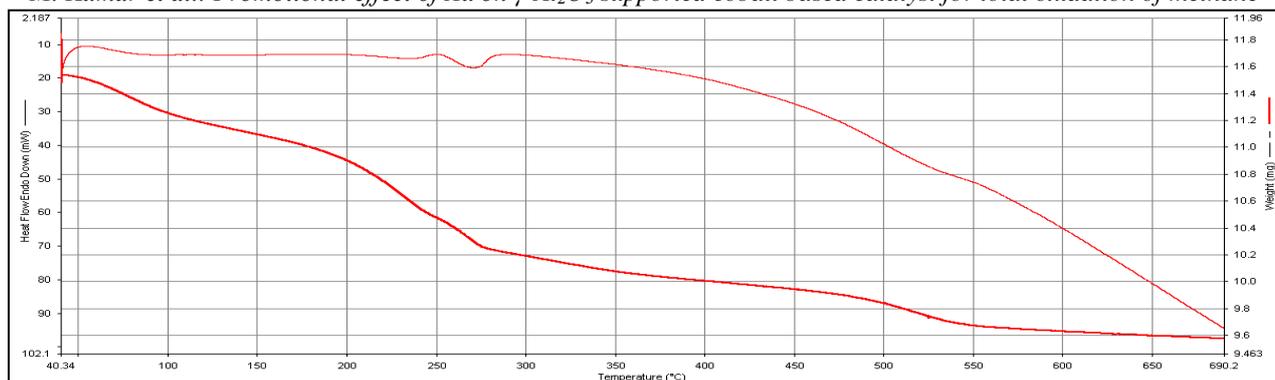
Figure 6. Graphical representation of TGA/DSC thermograms of the precursors of 13.45wt.% Au supported on 18wt.%Co/γ-Al₂O₃
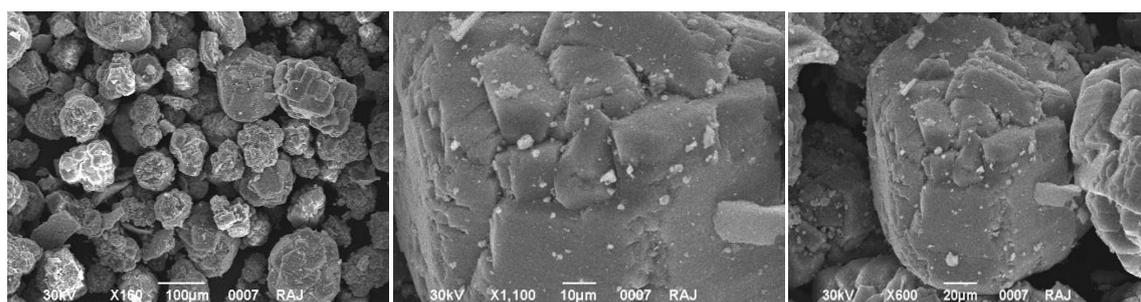

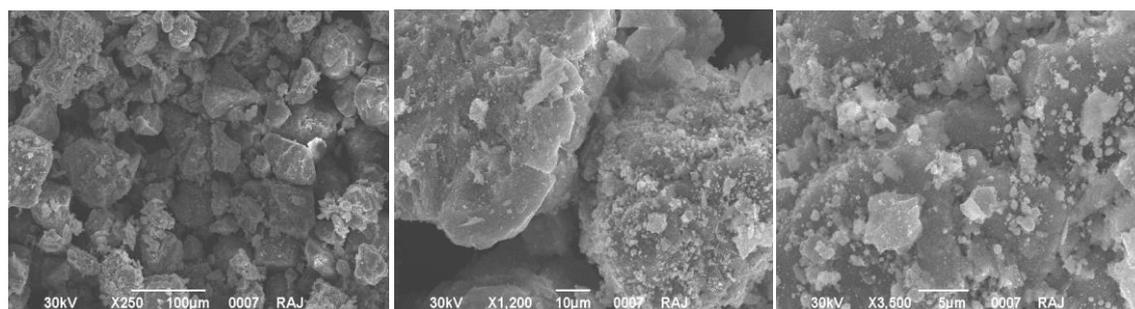**Figure 7.** SEM micrographs of G51 catalyst.

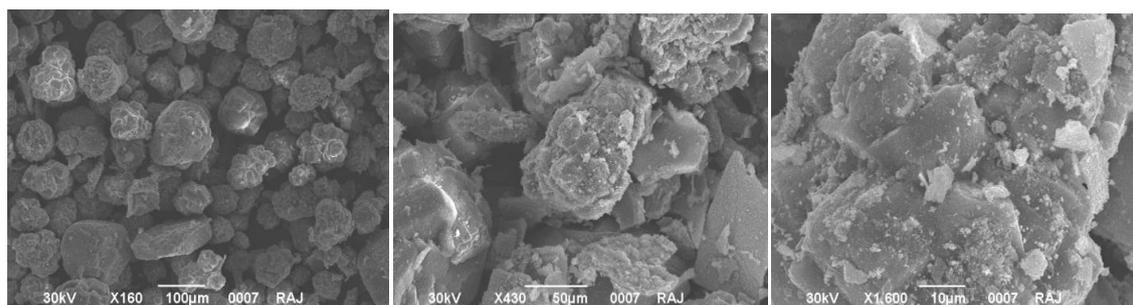**Figure 8.** SEM micrographs of G52 catalyst.

Figure 9. SEM micrographs of G53 catalyst.

SEM analysis

The SEM micrographs of all catalysts are shown in figures 7-11. Figure 7 shows the G51 catalyst, the flakes and spheres of cobalt are dispersed on alumina; separate cobalt oxide particles are also present. It can be seen that the gold particles are also dispersed on cobalt oxide, as well as on alumina. Figure 8 represents the SEM micrographs of catalyst G52 at various resolutions. It can be seen that the density of the gold particles is very high in comparison to G51, which is due to the high concentration of gold in comparison to G51. Larger spheres of gold are dispersed on cobalt and alumina.
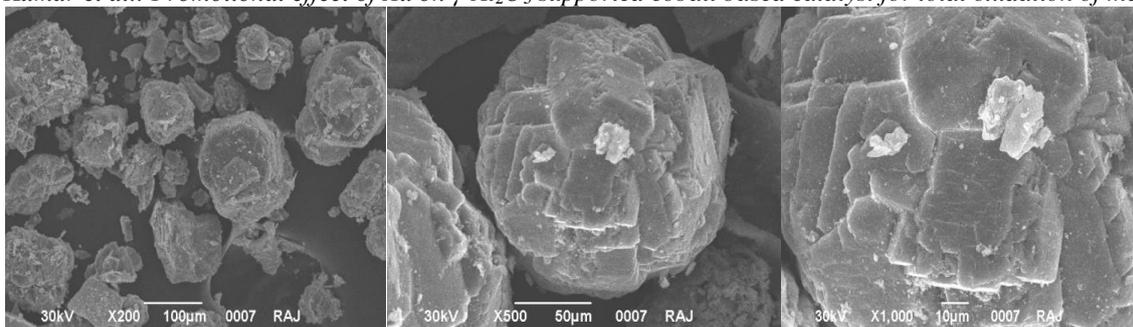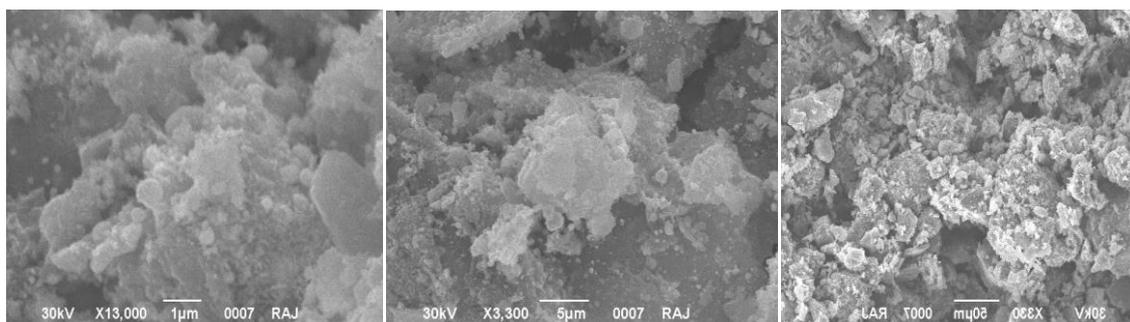
Figure 10. SEM micrographs of G54 catalyst**.**

Figure 11. SEM micrographs of G55 catalyst**.**

Figure 9 represents the SEM images of G53 catalysts. Huge blocks or particles of both gold and cobalt are dispersed on alumina which is in good agreement with the catalytic activity of the catalyst. The SEM micrographs of the highly active catalyst G54 are presented in figure 10. It can be seen that the gold particles are dispersed on cobalt species which are further embedded in alumina particles and this reflects the reason behind the high activity of the catalyst. This morphology helps in oxidation/ combustion of methane and also provides very high surface area per gm of the catalyst. However, figure 11 shows that agglomeration of gold particles takes place due to their high concentration.

CONCLUSIONS

Six catalyst samples having different gold composition and a constant amount of cobalt and alumina were prepared by a deposition/ precipitation method and tested for methane oxidation. Table 3 represents the temperature required for {100, 50 and 10}% conversion of methane for each catalyst. The catalytic performance for the said reaction (1) and the morphology of the catalysts strongly depend upon the gold content in the catalyst. The activation energy required for reaction is different for each catalyst and is also a function of gold content. The catalyst having 13.45% gold shows the best catalytic performance, this is ascribed to the uniform dispersion of gold species in the catalyst. The catalyst sample having 4.06 wt.% gold exhibits poor activity. The catalyst without gold shows the worst activity among all catalysts. Figure 12 shows the promotional effect of gold. It is clearly seen that light off temperature is tremendously increased with addition of gold. The catalytic activity of the tested samples is as follows: Au (13.45>10.44>16.27>07.21>04.06>0 wt. %) - CoO$_x$/γ-Al$_2$O$_3$. All six catalysts are active for CH$_4$ oxidation and do not show deactivation of catalytic activity for 50 h of continuous run at 380 °C at different levels of methane conversions.
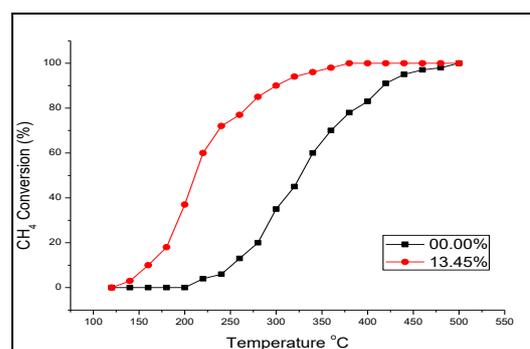

Figure 12. Promotional effect of gold (catalyst with 0, 13.45% Au) on methane conversion.

Table 3. Promotional effect of Au loading on light off temperatures of different catalysts prepared by a deposition precipitation method

Catalyst ID	Gold (wt.%)	Co:Au	Temperature (°C)		
			T$_{10\%}$	T$_{50\%}$	T$_{100\%}$
G50	00.00	5:0	260	350	490
G51	04.06	5:1	245	325	460
G52	07.21	5:2	270	345	450
G53	10.44	5:3	195	240	400
G54	13.45	5:4	180	215	380
G55	16.27	5:5	220	280	415

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ПРОМОЦИОНАЛЕН ЕФЕКТ НА Au ВЪРХУ γ -Al₂O₃ НАНЕСЕН КАТАЛИЗАТОР НА БАЗАТА НА КОБАЛТ ЗА ПЪЛНО ОКИСЛЕНИЕ НА МЕТАН

М. Кумар[1,2]*, Г. Ратан[1], Р. Прасад[3]

¹Институт по инженерна химия и технология „Д-р С. С. Бхатнагар", Панджабски университет, Чандигар 160014, Индия
²Департамент по инженерна химия, Университет на Чандигар, Мохали, Панджаб, Индия
³Департамент по инженерна химия, IIT-BHU, Варанаси, У.П., Индия

(Резюме)

Изучено е влиянието на съдържанието на злато в интервала от 4 мас. % - 16 мас. % върху отнасянията на катализатора Co/γ-Al₂O₃ при окислението на CH₄. Промотирани със злато CoOₓ/γ-Al₂O₃ катализатори са получени чрез утаително нанасяне и калциниране при 550ºC. При повишаване на съдържанието на злато се наблюдава значително повишаване на активността ($T_{100\%}$=380 ºC) при окисляване на CH₄, като при около 13.45 тегл. % Au се достига до плато. Каталитичната активност е изучена при постоянно тегло на катализатора (500 mg) в поток от въздух, съдържащ 1 % CH₄, внасян в реактора със скорост от 150 mL/min. Катализаторите са охарактеризирани чрез XRD, TGA/DSC и SEM. Каталитичната активност на катализаторите е в реда: Au (13.45>10.44>16.27>07.21>04.06>00.00 тегл. %)-CoOₓ/γ-Al₂O₃. Установено е, че катализаторът, съдържащ 13.45 тегл. % Au, нанесено върху 18 wt. % Co/γ-Al₂O₃ е най-активен и понижава температурата ($T_{100\%}$=380 ºC) на редукция на метан със 110ºC в сравнение с катализатор несъдържащ злато, т.е., 18 тегл.% Co/γ-Al₂O₃ ($T_{100\%}$=490 ºC).