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Research Article

# Total Oxidation of CO Using Cu & Co Catalyst: Kinetic Study and Calcinations Effect

**Gaurav Rattan***, **Rajwant Kaur**

*Dr. S. S. Bhatnagar University Institute of Chemical Engineering & Technology, Panjab University, Chandigarh, India*

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## Abstract

The present study deals with the oxidation of CO using base metal catalysts. A series of copper cobalt with different mole ratios were prepared by wet-impregnation method calcined at 400 °C for 3 h for the oxidation of CO. The mole ratios were varied from 1:1 to 1:5 by varying the weight of nitrates accordingly. It was found that 1:3 and 1:4 are active catalyst among the other prepared catalysts. Further, the two catalysts (1:3 & 1:4) were prepared by co-precipitation and citric sol-gel methods in order to see the effect of preparation method. The results showed that the catalyst prepared by co-precipitation method is good in terms of activity for CO oxidation. The best selected catalyst was characterized by TGA/DSC and XRD. Kinetic study was also performed on the selected catalyst. © 2015 BCREC UNDIP. All rights reserved.

***Keywords***: CO Oxidation; Catalyst; Copper; Cobalt; Kinetics

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## 1. Introduction

The exploration and development of the catalyst for oxidation of CO has started earlier in the 19th century during the World War II from now. Huge literature has been accumulated on the catalyst development. The catalysts used are noble metal catalysts [1,2], gold based catalysts [3-5], Transition base metal catalysts [6,7] and provoskites. Due to urbanisation there is a rapid growth of vehicles around the globe and hence the air pollution associated with it. Carbon monoxide, hydrocarbons and oxides of nitrogen are some of the main pollutants associated with the vehicular exhaust. Carbon monoxide is known a silent killer among all these pollutants. As CO combines with haemoglobin to make carboxyhemoglobin which reduces the oxygen carrying capacity of blood and affect the human body. Therefore the removal of CO from the vehicular exhaust is challenging task. Noble metal based catalysts are very successful and proved to be paramount in vehicular industry. They are used in vehicular industry controlling the vehicular exhaust from many decades. The noble metals used are Platinum, Palladium and Rhodium for the removal of harmful products to less harmful products. However in terms of economy and their rare availability challenges the researchers and scientist to make some other alternative to remove all these pollutants effectively. Further, the regulations and legis-

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* Corresponding Author.
E-mail: grattan@pu.ac.in (Gaurav Rattan)
Telp: +91-8288071498

lation limits adopted by government on the vehicles is very stringent and it will be more stringent in the near future. This motivates the researchers around the globe to explore and developed such catalyst which can do the same job as that of the noble metal.

Transition base metal catalysts [8, 9, 16] have been studied in this regard from the past century and various techniques or catalyst have been developed which nearly do the same job as that of noble metal. One of the biggest advantages of base metal catalyst is there low cost and easy availability. Cobalt and copper are the two transition base metals [14, 20, 21] which are studied thoroughly for CO oxidation.

Xie *et al.* [15] Reported the preparation of $Co_3O_4$ nanorods by a morphology controlled method the resulting materials were steadily

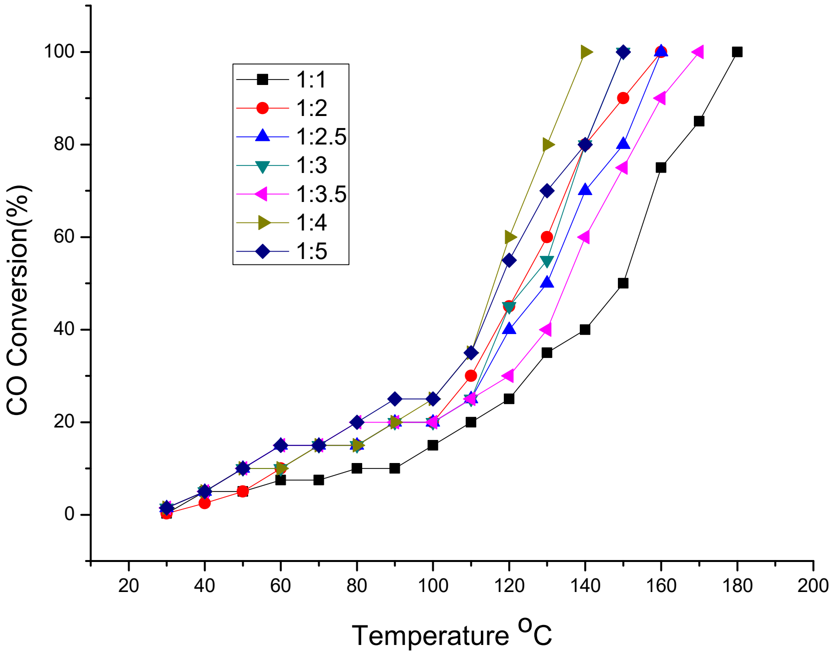

**Figure 1.** Catalytic activity of the catalyst by wet-impregnation method at different Cu:Co ratios

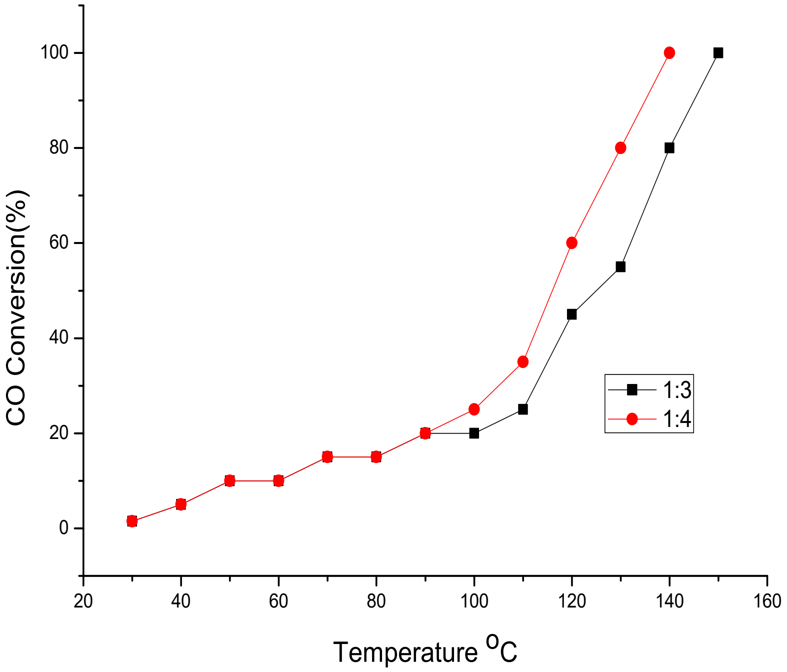

**Figure 2.** Comparison between mole ratio of Cu:Co 1:3 and 1:4 catalysts prepared by wet impregnation method

active for CO oxidation at as low as -77 °C. Feng *et al.* [15] reported that using facial pyrolysis method Mesoporous structured spinel $Co_3O_4$ could be fabricated and full CO conversion could be gotten at -70 °C. Yung *et al.* [17] studied cobalt based catalyst supported on zirconia for CO oxidation and it was reported by them that CO oxidation can takes place at 135 °C temperature. Further the characterization of the catalyst was done by TGA/DSC, Laser Raman spectroscopy and XPS. .

Recently Yuan *et a*l. [18] studied CO oxidation on cobalt based catalyst. In this paper, mesoporous spinel-type complex oxides $MCo_2O_{4(4.5)}$ were prepared using one-pot strategy from the single phase multi-component oxalate precursors. The prepared material showed high surface area and excellent catalytic activity for low temperature CO oxidation. The catalytic experiments indicated that full CO conversion could be achieved at as low as 30°C.

Copper is also studied for CO oxidation by numerous researchers [10, 19]. Guan *et al.* [10] studied copper-based catalyst and reported that complete conversion of CO takes place at 95 °C. The effect of activation temperature and initial CO concentration on CO oxidation over $Cu/TiO_2$ were further investigated. XRD, TEM, XPS-AES, $H_2$-TPR and FTIR of CO adsorption were employed to characterize $Cu/TiO_2$ samples and the exact composition of $Cu/TiO_2$ prepared by photo-deposition was determined to be Cu-$Cu_2O/TiO_2$.

The combination of cobalt and copper is also depicted in many research articles [11-13]. However, these combination is not studied thoroughly. The combination of these two base metals can be studied thoroughly and interesting results could be there. Therefore, the present article deals with the combination of these two base metals catalyst and the optimisation were evaluated in terms of catalytic activity of CO. The same catalyst was characterized by TGA/DSC and XRD. Moreover, the kinetic study has been done on the best selected catalyst.

## 2. Experimental Section

### 2.1. Preparation of the Catalysts

A number of cobalt and copper based catalysts was prepared using its nitrates salts with different mole ratios such as 1:1, 1:2, 1:2.5, 1:3, 1:3.5, 1:4, and 1:5. The entire chemicals used were of AR grade. The copper nitrate trihydrate and cobalt nitrate hexahydrate salts were used to prepare the catalyst for CO oxidation. The catalyst was prepared by three different methods, namely: (i) wet-impregnation method, (ii) sol-gel method, and (iii) co-precipitation method. The mole ratio of copper cobalt varies at different composition from 1:1 to 1:5. The prepared catalysts were stored in an air tight bottle (100–120 mesh size particles). Preparation methods of the catalyst are described as follow:

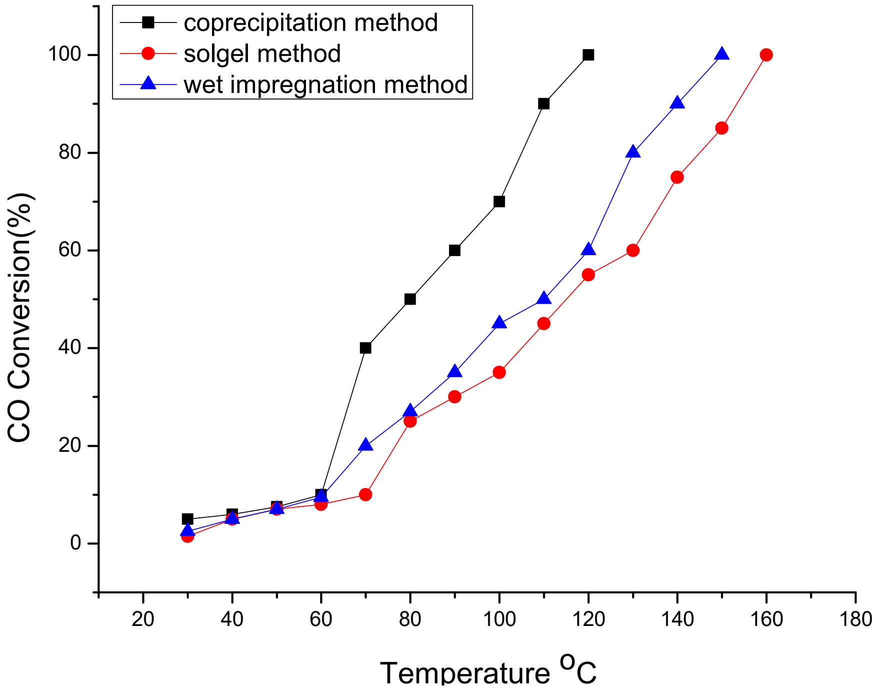

**Figure 3.** Effect of the preparation method on catalyst with mole ratio of Cu: Co 1:3

**Wet-impregnation method [12]:** the stoichiometric amounts of copper cobalt nitrates salt were dissolved in distilled water under continuous stirring and heating at 80 °C till dryness appear. Samples were further dried in an oven overnight at 100 °C and calcined in a muffle furnace at 400 °C for 3 h.

**Citric acid Sol-gel method [19]:** metal nitrates were dissolved in distilled water according to required molar ratio and citric acid was added as a complexing agent with 1.3:1 ratio of acid to metals ions. Polyethylene glycol was added as an additive with 10% of the citric acid. The blended solution was mixed under continuous stirring at 80 °C till a gel was formed. This gel was dried overnight in oven at 100 °C and calcined in the muffle furnace at 400 °C for 3 h.

**Co-precipitation method [14]:** the solution of metal nitrates was slowly added to the solution of sodium carbonate in distilled water under continuous stirring by maintaining the pH 10. After some time precipitates formed were filtered with Whatman's filter paper and washed several times with distilled water and dried in an oven overnight at 100 °C. After drying, the sample was calcined in muffle furnace at 400 °C for 3 h.

### 2.2. Catalytic Activity Measurements

The catalytic activity was measured in tubular packed bed reactor at atmospheric pressure. 100 mg of the prepared catalyst was diluted or mixed with the 5 mL of alumina [22]. The mixture prepared was placed in fixed bed down flow tubular reactor by making bed of glass wool. The reactor was placed vertically in a open-split furnace. The reactant consist of 2% of CO was mixed with air having a total flow rate of 100 mL/min. The flow rate of CO and air were monitored with the help of digital flow meters. The air feed was made free of moisture and $CO_2$ by passing it through CaO and KOH pellet. The steady state activity was measured from room temperature to 250 $^0$C. The catalytic experiments were carried out under steady state conditions. The reactants and products were analysed under steady state conditions for CO and $CO_2$ with the help of Gas Chromatograph. The catalytic activity was expressed by the conversion of CO calculated by Equation (1).

$$X_{CO} = \frac{\left(C_{CO,in} - C_{CO,out}\right)}{C_{CO,in}} \quad (1)$$

## 3. Results and Discussions

### 3.1. Catalysts Prepared by Wet Impregnation Method

The catalytic activity of the catalyst prepared by wet-impregnation method is shown in figure 1. The mole ratio of Cu:Co are 1:1, 1:2, 1:2.5, 1:3, 1:3.5, 1:4, and 1:5. The various mole ratio of copper cobalt gives the CO conversion at different temperature. It is clearly shown in the graph that the catalytic activity increase from mole ratio Cu:Co of 1:1 to 1:5 for most of the cases. It is clearly visible in figure that the catalyst having mole ratio of Cu:Co of 1:3 and 1:4 respectively gives better result in terms of activity. The mole ratio of Cu:Co of 1:3 and 1:4 gives 100% CO oxidation at minimum temperature. Catalyst having mole ratio of 1:3 gives 100% CO conversion at 150 $^o$C and at 1:4 gives 100% CO conversion at 140 $^o$C which is the minimum temperature as compared to the other prepared catalysts. In order to see the Conversion versus temperature of mole ratio of copper cobalt 1:3 and 1:4 clearly shown in Figure 2.

### 3.2. Effect of The Preparation Method on Catalytic Activity

From the previous result of the catalyst prepared by wet-impregnation method find that

**Table 1.** Light off temperatures of Cu:Co 1:3 prepared by different methods

| Preparation method | Temperature ($^o$C) | | |
|---|---|---|---|
| | $T_{10\%}$ | $T_{50\%}$ | $T_{100\%}$ |
| Co-precipitation method | 40 | 80 | 120 |
| Sol-gel method | 50 | 120 | 160 |
| wet-impregnation method | 40 | 110 | 150 |

**Table 2.** Light off temperatures of Cu:Co of 1:4 prepared by different methods

| Preparation method | Temperature ($^o$C) | | |
|---|---|---|---|
| | $T_{10\%}$ | $T_{50\%}$ | $T_{100\%}$ |
| Co-precipitation method | 40 | 80 | 110 |
| Sol-gel method | 50 | 100 | 130 |
| wet-impregnation method | 60 | 110 | 140 |

1:3 and 1:4 is best active catalyst. To see the effect of preparation methods the mole ratio of copper cobalt catalyst were prepared by other two methods which are sol-gel and co-precipitation method.

### 3.2.1. Catalysts Having Mole Ratio of Cu:Co=1:3

Co-precipitation method gives best result as compared to the catalyst prepared by other method. The catalyst prepared by co-precipitation method gives 100% CO conversion at 120 °C. However the catalyst prepared by sol-gel method is poorest in terms of activity. Table1. gives light off temperature of catalyst having Cu:Co=1:3.

### 3.2.2. Catalytic Activity of Catalysts with Molar Ratio of Cu:Co=1:4

In this, catalyst with mole ratio 1:4 prepared by co-precipitation method gives good result as compared to the other methods. The catalyst prepared by co-precipitation method gives 100% CO conversion at 110 °C which is the lowest from the other catalysts. While the catalyst prepared by sol-gel and wet- impregnation method showed relatively low active for CO oxidation and complete CO conversion achieved at 130 °C and 140 °C respectively. Table 2 gives light off temperature of catalyst having Cu:Co = 1:4. From Figure 5, it is clearly shown that the catalyst prepared by co-precipitation (Cu:Co=1:4) method is the best active catalyst for CO oxidation i.e. $T_{100\%}$= 110 °C. For light off temperature of Cu:Co = 1:3 and 1:4 is shown in Table 3.

### 3.2.3. Effect of Calcination Temperature

Effect of the calcination temperature on the best active catalyst is studied by varying the calcinations temperature, i.e. from 300 to 500 °C. The calcined catalysts were studied for CO oxidation and the results are obtained in Figure 6. It can be seen from the figure that the catalyst calcined at 400 °C exhibited the highest activity and achieved 100% CO conversion at 110 °C. Calcination of the catalysts at higher temperature caused sintering, which led to a decrease in the surface area and decrease in catalytic activity [22].

## 3.3. Characterization

### 3.3.1. Thermo Gravimetric Analysis (TGA/DSC)

In the present work TGA was carried out for uncalcined sample prepared by co-precipitation

**Table 3.** Light off temperatures of Cu:Co of 1:3 and 1:4 prepared by co-precipitation method

| Co-precipitation method | Temperature (°C) | | |
|---|---|---|---|
| | $T_{10\%}$ | $T_{50\%}$ | $T_{100\%}$ |
| 1:3 | 40 | 80 | 120 |
| 1:4 | 40 | 70 | 110 |

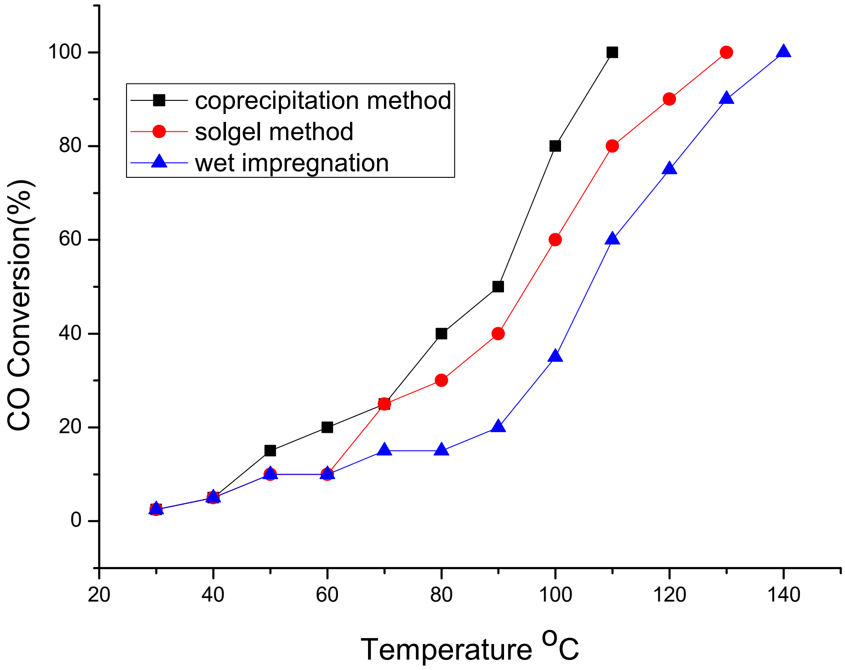

**Figure 4.** Effect on preparation method on mole ratio of Cu:Co 1:4

method. Thermo gravimetric analysis (TGA) is the most widely used thermal method based on the measurement of mass loss of material as a function of temperature. In thermogravimetry a continuous graph of mass change against temperature is obtained when a substance is heated at uniform rate or kept at constant temperature. A plot of mass change versus temperature (*T*) is referred to as the thermo gravimetric curve (TG curve). For the TG curve, we generally plot mass (*m*) decreasing downwards on the *y* axis (ordinate), and temperature (*T*) increasing to the right on the x axis (abscissa) as shown in Figures 7 and 8.

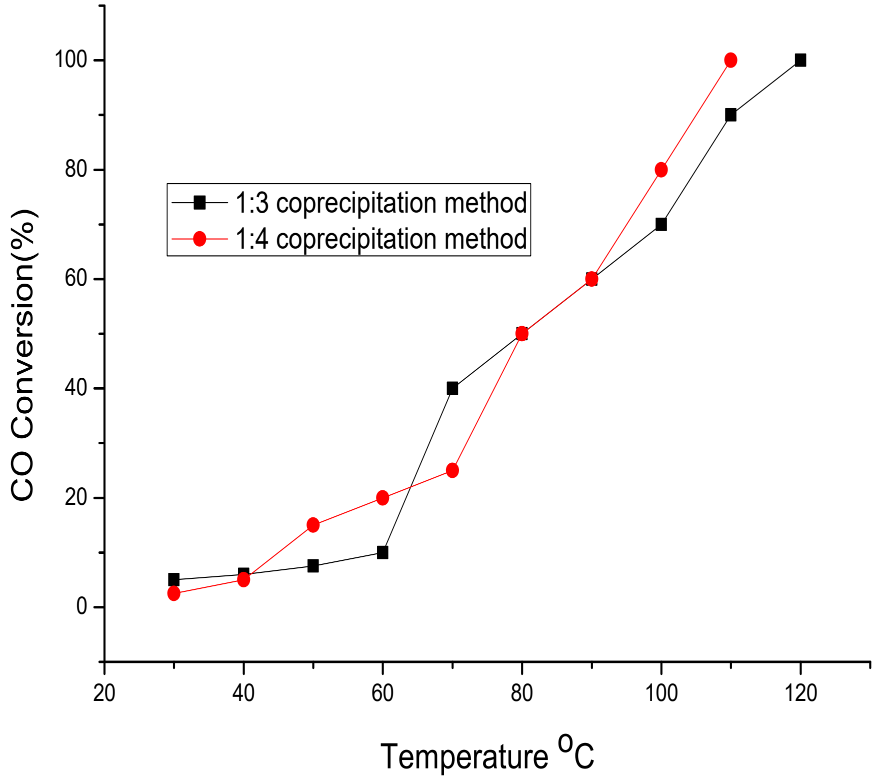

**Figure 5.** Comparison between mole ratio of Cu:Co of 1:3 and 1:4 prepared by co precipitation method

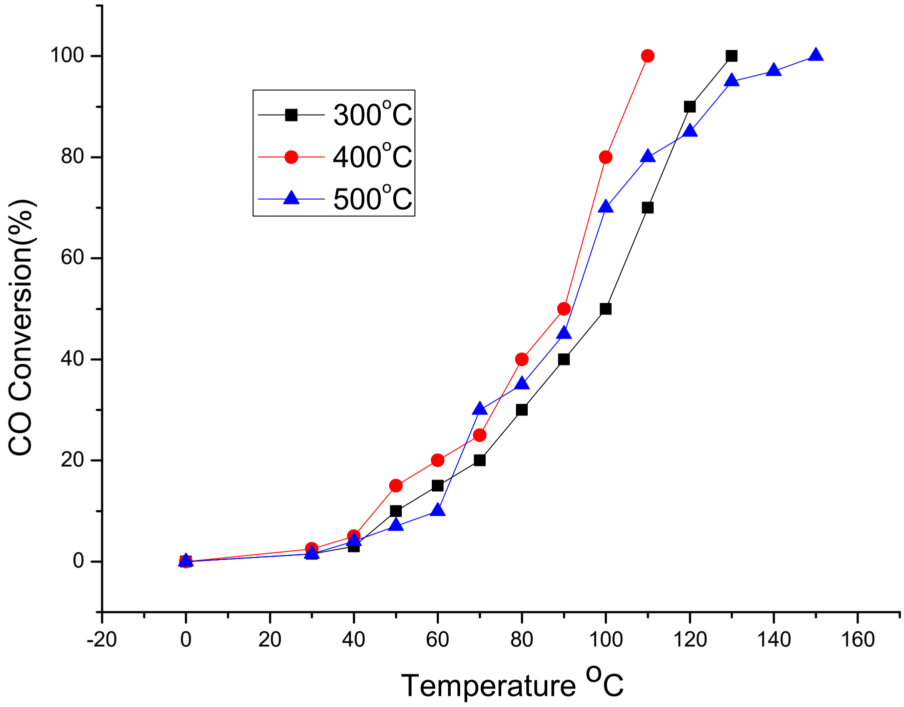

**Figure 6.** Effect of the calcinations temperature on mole ratio of Cu:Co 1:4

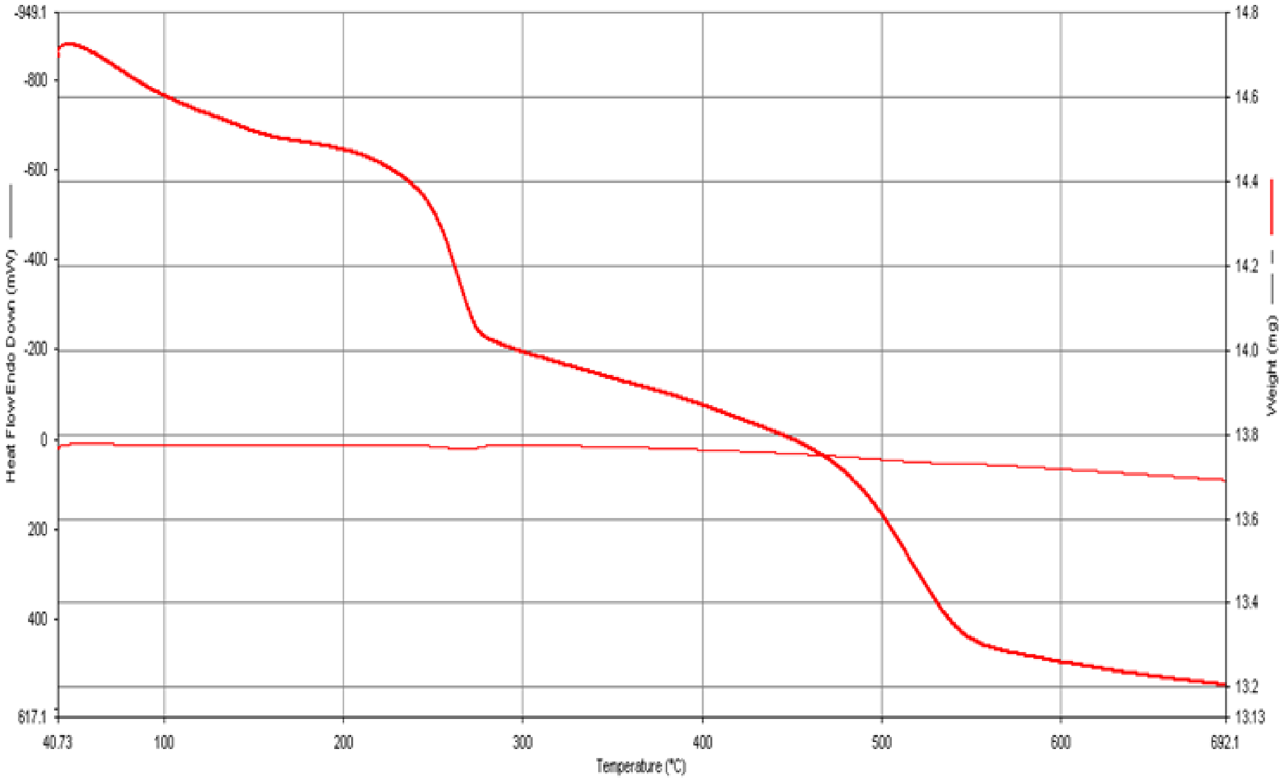

**Figure 7.** TGA/DSC curve of the catalysts having molar ratio Cu:Co of 1:3 prepared by co-precipitation method

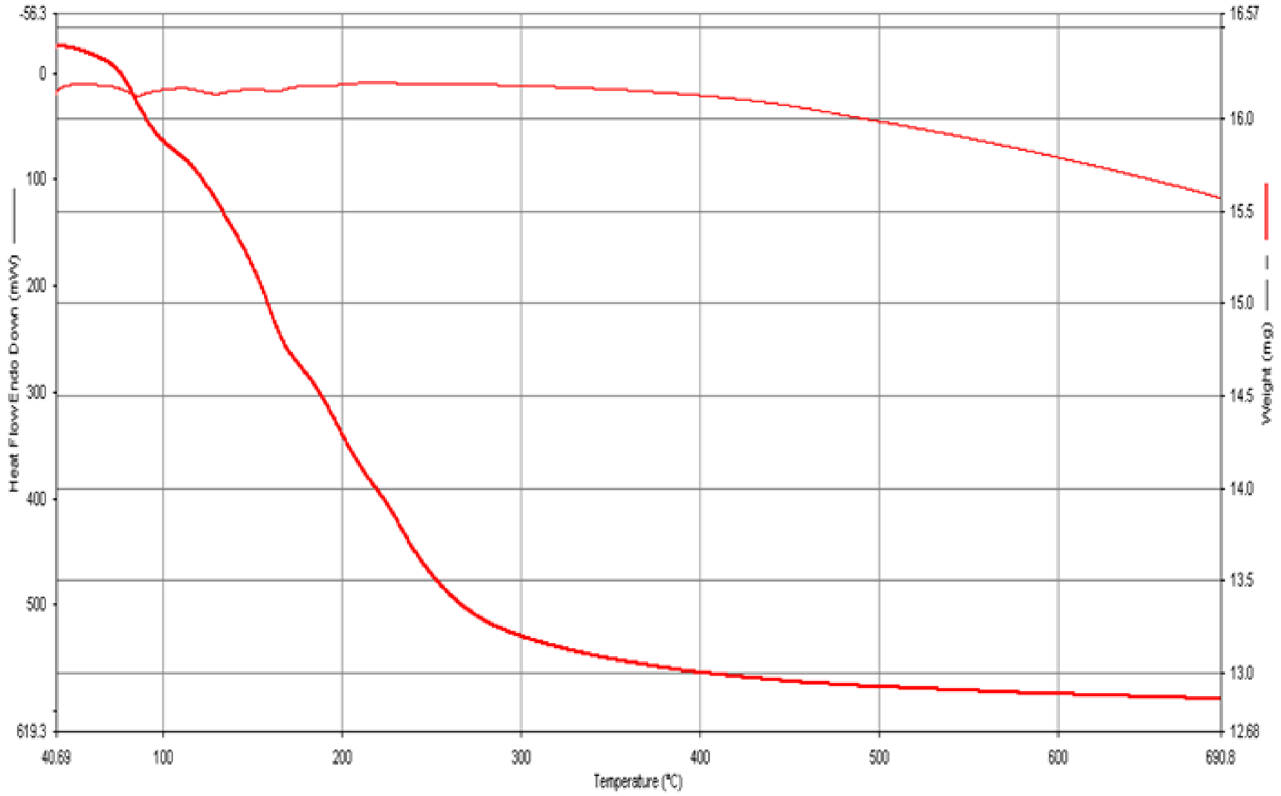

**Figure 8.** TGA/DSC curve of the catalysts having molar ration Cu:Co of 1:4 prepared by co-precipitation method

TGA/DSC curve of the catalysts having molar ratio of copper cobalt 1:3 and 1:4 prepared by co-precipitation method is shown in Figures 7 and 8, respectively. In the Figure 7 temperature range of 90 to 200 °C depicts the moisture content loss and the main weight lost in between 300-600 °C. This may be due to the decomposition of the various nitrates present in the catalyst. However there is a sharp decrease in the weight of the catalyst from 100 to 400 °C in Figure 8. The moisture content may be attributed to the temperature of 100 to 200 °C. However the rest is because of the decomposition of metal ions and nitrates present in the catalyst. The total weight loss is 20-25% as shown in figure.

### 3.3.2. X-Rays Diffraction

X-Ray diffraction (XRD) of the catalyst samples was carried out to indentify the phases and oxidation states present in the catalysts. XRD pattern was recorded in X'PERT PRO diffractometer using CuKα radiation source with operating current and operation voltage were 40 mA and 45 KV respectively. The scanning range $2\Theta$ was 5.00 - 99.98° with divergence slit of 0.8709°. The continuous scanning was done with step size of 0.0170° and with scan step time of 30.36 s. The XRD pattern of the prepared mole ratio of copper cobalt catalysts 1:3 and 1:4 calcined at 400 °C. In the Figure 9 the representative XRD pattern in which peaks at $2\theta$ of 31.245, 36.758, 38.44, 44.465, 59.165, and 64.562 shows different lattice planes. Further the strong peak at 38.44 attributed to the cobalt species and rest of the peaks are due to the formation of oxides. The diffraction peaks of catalyst showed that the catalyst is almost amorphous. The broad diffraction peaks are due to the formation of small crystallites.

**Table 4.** Optimisation of calcination temperature Cu:Co of 1:4 between 300-500 °C

| Calcination Temperature (oC) | $T_{10\%}$ | $T_{50\%}$ | $T_{100\%}$ |
|---|---|---|---|
| 300 | 50 | 90 | 130 |
| 400 | 40 | 80 | 110 |
| 500 | 60 | 100 | 160 |

**Table 5.** Space velocity of 100 mg catalyst

| Sr.no | CO (ml/min) | Air (ml/min) | $W/F_{CO}$ (gcat.h/gmol) |
|---|---|---|---|
| 1 | 4 | 96 | 10.05 |
| 2 | 3 | 97 | 13.40 |
| 3 | 2 | 98 | 20.11 |
| 4 | 1 | 99 | 40.22 |

**Table 6.** Space velocity of 200 mg catalyst

| Sr.no | CO (ml/min) | Air (ml/min) | $W/F_{CO}$ (gcat.h/gmol) |
|---|---|---|---|
| 1 | 4 | 96 | 20.11 |
| 2 | 3 | 97 | 26.66 |
| 3 | 2 | 98 | 40.41 |
| 4 | 1 | 99 | 81.27 |

**Table 7.** CO conversion at different space velocity

| $W/F_{CO}$ | Conversion Temperature (°C) | | | |
|---|---|---|---|---|
| (gcat.h/gmol) | 30 | 40 | 50 | 60 |
| 10.05 | 0.016 | 0.027 | 0.042 | 0.050 |
| 13.40 | 0.025 | 0.040 | 0.055 | 0.070 |
| 20.11 | 0.041 | 0.057 | 0.072 | 0.085 |
| 40.22 | 0.058 | 0.075 | 0.090 | 0.100 |

**Table 8.** Fractional conversion of CO ($x_{CO}$) vs $W/F_{CO}$ at different temperature

| Temperature (°C) | $x_{CO}$ | ln $r_{obs}$ | ln $C_{co}$ |
|---|---|---|---|
| 30 | 0.016 | -6.14 | -10.10 |
| 30 | 0.025 | -6.16 | -10.11 |
| 30 | 0.041 | -6.2 | -10.13 |
| 30 | 0.058 | -6.24 | -10.15 |
| 40 | 0.027 | -5.71 | -10.11 |
| 40 | 0.040 | -5.75 | -10.13 |
| 40 | 0.057 | -5.78 | -10.15 |
| 40 | 0.075 | -5.79 | -10.16 |
| 50 | 0.042 | -5.49 | -10.13 |
| 50 | 0.055 | -5.495 | -10.14 |
| 50 | 0.072 | -5.53 | -10.16 |
| 50 | 0.090 | -5.55 | -10.18 |
| 60 | 0.050 | -5.18 | -10.14 |
| 60 | 0.070 | -5.22 | -10.16 |
| 60 | 0.085 | -5.224 | -10.18 |
| 60 | 0.100 | -5.27 | -10.19 |

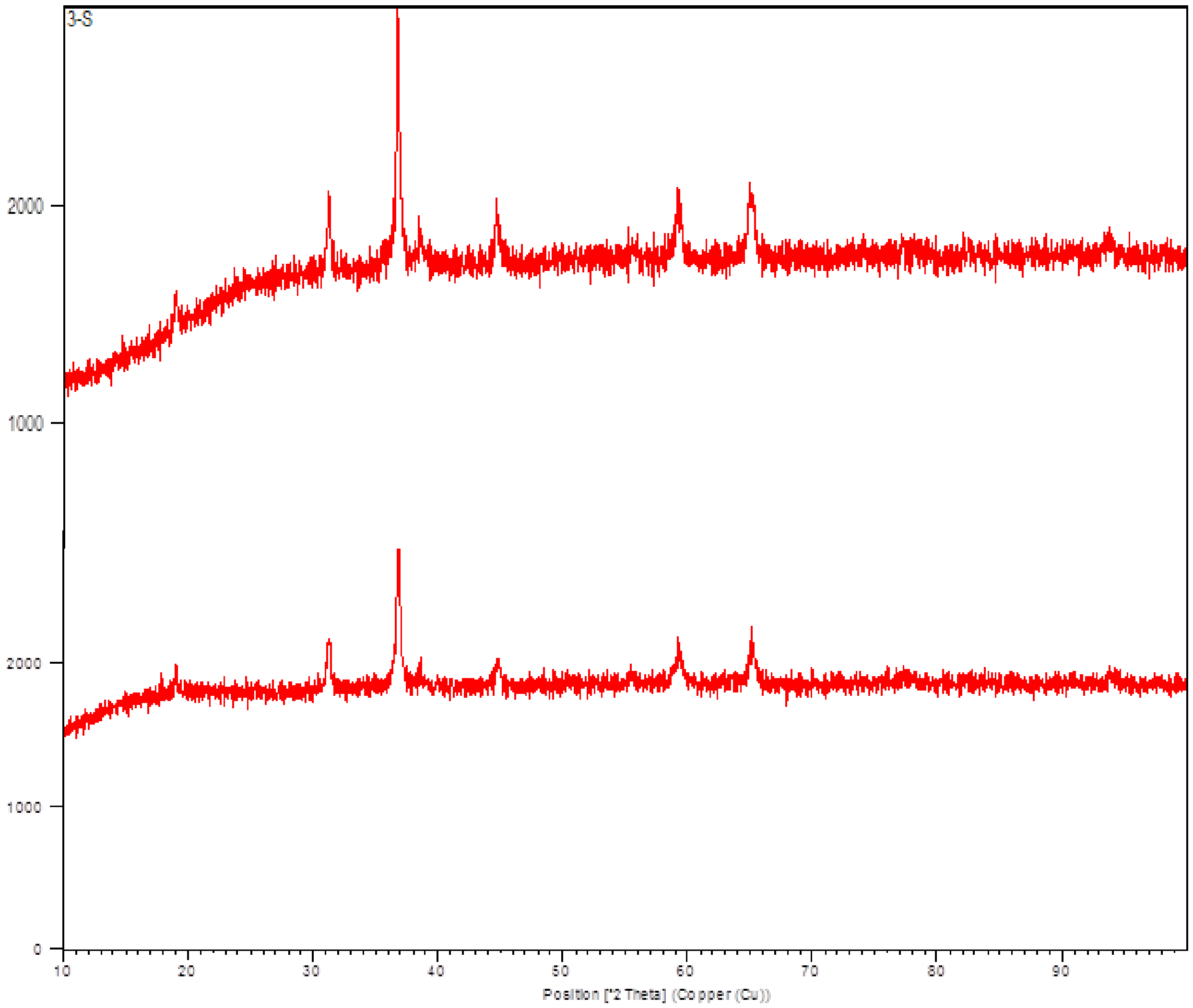

**Figure 9.** XRD pattern of catalysts having molar ratio of Cu:Co of 1:3 and 1:4

### 3.4. Kinetic Study

Chemical kinetics is also known as reaction kinetics, reaction kinetics is the study of rates of chemical processes. Chemical kinetics include investigation of how different experimental conditions can influence the speed of a chemical reaction and the yield information about the reaction's mechanism and transition states, as well as the construction models that can describe the characteristics of the chemical reaction. Chemical kinetics deals with the experimental determination of reaction rates from which rate laws and rate constants are derived. Relatively simple rate laws exist for zero order reactions, first order reactions, and second order reactions, and can be derived from others. The activation energy for a reaction is experimentally determined through the Arrhenius equation; the main factors that influence the reaction rate include the physical state of the reactants, the concentrations of the reactants, the temperature at which reaction occurs.

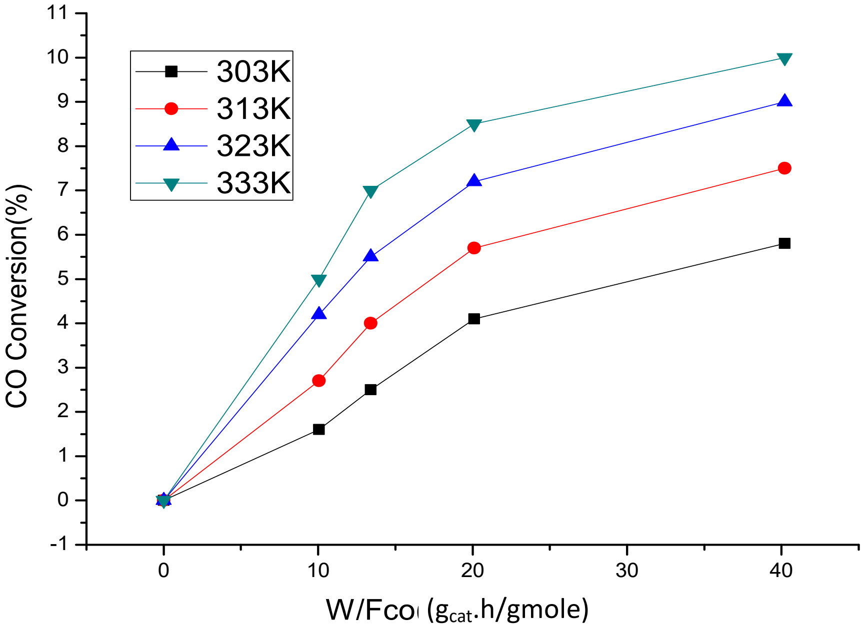

**Figure 10.** Plot of CO conversion vs. W/Fco at different temperature

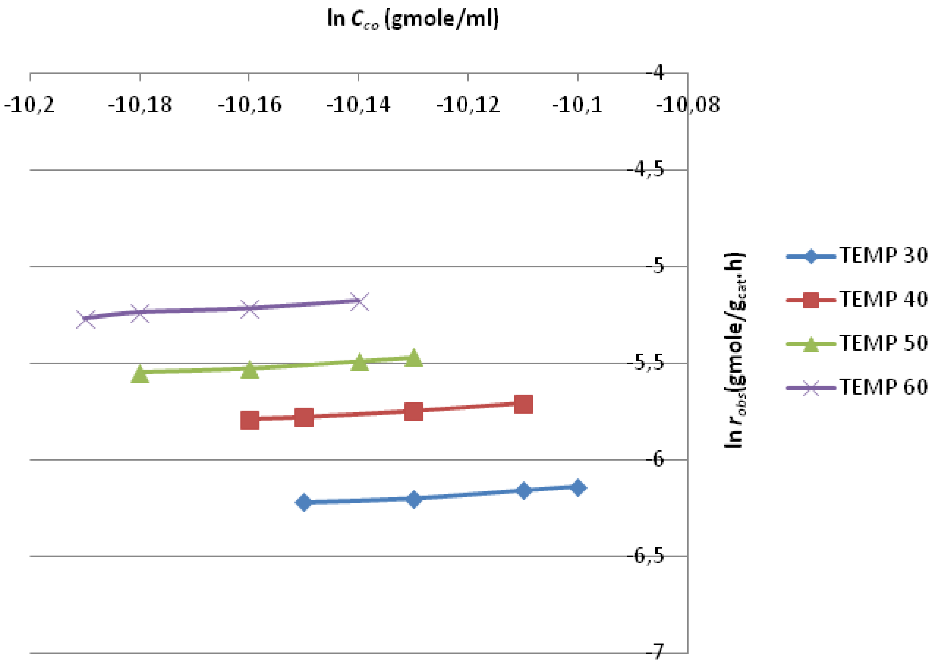

**Figure 11.** Plot of ln $r_{obs}$ vs. ln $C_{co}$ at different temperature ($^{o}$C)

### 3.4.1. Effect of The Space Velocity

To study the effect of space velocity on conversion of CO activity of different catalyst were examined with increasing the space velocity. The temperature and feed composition were fixed. The effect of space velocity on CO conversion is shown below in Table 5 and 6.

The Equation (2) can be obtained by performing material balance on CO in the reactor under steady state condition.

$$r_{obs} = \frac{dx_{CO}}{d(W/F_{CO})} \tag{2}$$

A series of experimental run in packed bed column was made using varying reciprocal weight hourly space velocity ($W/F_{CO}$). The mass of the catalyst, 100 mg diluted with 5 ml of alumina was kept constant throughout the steady state experimental program. The external and internal mass transfer effects were absent during the experiment. The variation in $W/F_{CO}$ was achieved by changing the feed rate of CO. To minimize the heat and mass transfer effect and to approximate differential reactor; conversion for each run was kept up to 10%. The rate at any conversion can be obtained by taking the slope of $x_{CO}$ versus $W/F_{CO}$ plot.

Once the rate of reaction is known as a function of concentration of reactants, an empirical power law type of Equation (3) can be fitted:

$$r_{obs} = k\, C_{O_2}^{m}\ C_{CO}^{n} \tag{3}$$

**Table 9.** Order of the reaction

| T (K) | 303 | 313 | 323 | 333 |
|---|---|---|---|---|
| *m* | 1.6727 | 1.610 | 1.627 | 1.687 |
| ln *K* | 10 | 10.48 | 11 | 11.83 |

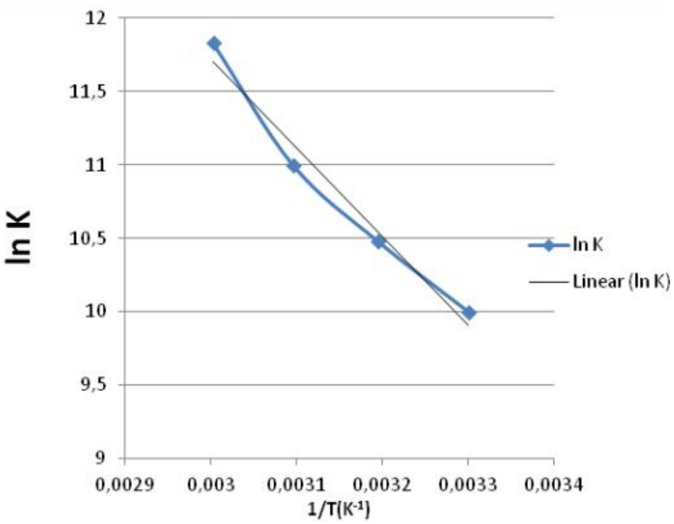

**Figure 12.** Arrhenius plot ln *K* versus 1/*T* ($k^{-1}$)

In the present study oxygen was in excess, therefore the rate expression reduces to Equation (4):

$$r_{obs} = k\, C_{CO}^{n} \tag{4}$$

The *k* is a function of temperature and can be represented by Arrhenius in Equation (5).
Taking natural log on both sides of the above equation, we get Equation (5) of straight line.

$$ln\ (rate) = ln\ K + n\ ln\ (C_{CO}) \tag{5}$$

where:

$$K = A\ exp\ (-E/RT) \tag{6}$$

If the power law functionality is successful and *r* versus $C_{CO}$ data are known at constant temperature, *n* and *k* at that temperature can be determined from log-log plot of $r_{obs}$ versus $C_{CO}$.

Linear plot were obtained when ln $r_{obs}$ and ln $C_{CO}$ were plotted. Slope and intercept of the lines gives order of reaction and ln *k*, respectively. The reaction rate constant follows Arrhenius law. The activation energy of the reaction and frequency factor *A* can be evaluated from the slope of a plot of ln *k* versus 1/*T*.

A plot of CO conversion to $CO_2$ ($x_{CO}$), when the catalyst showed steady performance, versus $W/F_{CO}$ at four different temperatures is shown in Figure 10. Rate of reaction at different conversion for each temperature can be calculated by measuring the slope of the curve. The plot of ln $r_{obs}$ and ln $c_{co}$ gives the linear plot. The intercept and slope of this curve gives value of ln *k* and order of reaction, respectively. The slope and intercept of the Figure 11 gives order of reaction (*m*) and ln *K*, respectively. Slope and intercepts of the line give order of the reaction (*m*) and ln *K* respectively, which are reported in Table 9. Order of the reaction was found to be 1.6. The activation energy (*E*) and pre-exponential factor (*A*) of Arrhenius equation were calculated by measuring the slope and intercept respectively of the plot of ln *K* versus 1/*T* as shown in Figure 12. The values obtained are: (i). Activation energy (E) = 502.03 kJ $mole^{-1}$, (ii). Pre-exponential factor (A) = 9.1064 x $10^{12}$. Rate of reaction as presented in Equation (7) was found in temperature range of 303- 333 K.

$$Rate = 9.1064x10^{12}\ \exp(502.03/RT)(C_{CO})^{1.6} \tag{7}$$

## 4. Conclusion

The mole ratios of Cu:Co prepared by co-precipitation method have higher activity than the catalyst prepared by wet impregnation and sol gel methods. The catalyst were calcined at 400 $^0$C and activity was observed for CO oxidation it was seen that each catalyst containing Cu:Co of 1:4 have higher activity. The catalytic test shows that the mole ratio of Cu:Co of 1:4 exhibits the highest activity towards CO oxidation at lowest temperature 110 °C. The best activity of the prepared 1:4 catalyst calcined at 400 °C.

The kinetic study was done by taking the catalyst having mole ratio of Cu:Co of 1:4. This was prepared by co-precipitation method. Effect of space velocity was observed at four different temperatures. The rate was obtained by taking the slope of $x_{CO}$ versus $W/F_{CO}$ plot. On the basis of experimental finding the order of reaction was observed 1.6 and the rate equation was found expressed as in Equation (7).

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