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RESEARCH ARTICLE

THE OXIDATION OF CARBON MONOXIDE BY USING DIFFERENT COBALT PRECURSORS PREPARED CATALYSTS

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ABSTRACT

The carbon monoxide is a toxic and life-threatening gas to humans and other forms of air-breathing life. In present time, the low-temperature oxidation of carbon monoxide is applicable in large scale of application in automobile and other fields. In this paper, the different types of cobalt precursors were prepared in the laboratory for oxidation of carbon monoxide at low temperature. The precursor was converted into the catalyst by calcination at 300°C temperature. The cobalt precursor first drying at 120°C temperature for 12h then calcined at 300°C in different calcination conditions. The catalytic activity measurements were examined in room temperature to 250°C, the 100mg of catalyst was used in presence of 2.5%CO with air and total flow rate was 60mL/min. They are several techniques has used for the characterization of the catalysts such as XRD, SEM-EDX, FTIR and XPS analysis. The low-temperature CO oxidation activity measurement showed in all of the catalyst and the best activity was obtained from cobalt oxalate precursor prepared catalyst at 110°C temperature. The order of activity for different cobalt precursor was as follows: Cobalt Oxalate > Cobalt Acetate > Cobalt Nitrate.

INTRODUCTION

The oxidation of carbon monoxide at low temperature by using catalyst is a very important topic in the current scenario; it is used in many applications like automotive, residential air cleaning technologies, gas masks for mining applications, CO detectors, and fuel cell also (Balikci and Guldur, 2007). The carbon monoxide combines with hemoglobin presence in blood cells and converted into carboxy-hemoglobin (CoHb), it decreases the oxygen carrying ability of the human body. The automobile engine is also a good source of CO in the environment, in comparison to a diesel engine, the petrol engine produces more carbon monoxide (Cholakov, 2000). A catalytic converter is a device it used in a vehicle for reducing the toxicity emissions from an internal combustion engine. In the presence of the catalyst, the rate of chemical reaction is increased; it acts like an agent that reduces the activation energy of reactions. On start-up car exhaust catalysts take a little time to become effective, and this primarily due to the time required to heat the catalyst using the exothermicity of the combustion reactions (Ghaffari *et al*., 2008). The different types of catalyst have a different property for oxidation of CO at a low temperature and the property of the catalysts was analyzed by different types of characterization. In the presence study, we have to use different types of cobalt oxide catalyst (CoOx) for oxidation of CO at low temperature. The cobalt precursors after calcination used as a catalyst for conversion of CO, the precursors are cobalt nitrate, cobalt oxalate, and cobalt acetate. The size and shape of a catalyst possess increased the number of active sites per unit mass of the catalyst material; it reduces the amount of catalyst especially (Janssens *et al*., 2007). The synthesized of unique nanorod-shaped cobalt oxide catalysts (Co_3O_4) that oxidized CO at temperatures as low as -77°C. In our best knowledge, we understand the role of different types of cobalt precursor to oxidation of CO is analysis by different types of characterization. The in-situ FTIR studies we understand the different transient conditions to gain insight into the reaction mechanisms (Mankidy *et al*., 2014). The different cobalt precursors like cobalt nitrate, cobalt acetate, and cobalt oxalate have a different pore structure and the size of pores is easily detected by XRD analysis. To increase the activity of CO oxidation, the cobalt is usually deposited on a high surface area support to achieve a high metal dispersion. The surface cobalt metal atoms are very active for CO oxidation (Panpranot *et al*., 2003). The main phase of cobalt in the oxidized catalyst is Co_3O_4 under the reaction conditions. In general, the catalytic properties depend on the reaction conditions, catalyst composition, metal dispersion and the types of inorganic support materials used

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(Rojanapipatkul *et al*., 2012). The Co_3O_4 and CoO are stable oxides among the cobalt oxides system. A valence of cobalt higher than +3 is thermally unstable (Lin *et al*., 2003). The Co_3O_4 is a spinel structure; CoO is a cubic structure and their structure stable in nature. The cobalt oxide is a versatile material among the transition metal oxides, it's used in the development of the rechargeable battery and CO sensors (Lin *et al*., 2003).

MATERIALS AND METHODS

Catalyst preparation

In the cobalt oxide catalyst, the precursor we used to the preparation of the catalysts are cobalt nitrate, cobalt acetate, and cobalt oxalate. The cobalt nitrate and cobalt acetate precursor were directly used as a catalyst after calcination, they are manufacturing of the Otto Chemie Company and all the chemicals used for the preparation of the catalysts were of A.R. grade. The cobalt oxalate precursor is prepared by the mixing of cobalt nitrate and oxalic acid (molar ratio 2:1) into the solutions. The precursor of cobalt nitrate, cobalt acetate, and cobalt oxalate was dried at 120°C for 12 hr into the oven. The calcination of the catalyst precursor was carried out in three ways; in the first we used the conventional method of calcination is stagnant air calcination (SAC) in the absence of air, second is flowing air calcination (FAC) in the presence of air and third reactive calcinations (RC) in the presence of CO and air at 400°C for 2h. The RC of the catalyst was carried out in situ in a compact bench scale fixed bed with a down flow tubular reactor. The calcination of the Cobalt precursors was performed under a reactive mixture of 4.5% of CO in air at 400°C temperature for 2 hours, just before the activity measurement of the catalyst.

Table 1. The nomenclature used for the catalyst samples in this study is as follows

Catalyst Name	Nomenclature
Cobalt Nitrate	CoN
Cobalt Acetate	CoAc
Cobalt Oxalate	CoO

Characterization

The X-ray measurement of the catalyst was done by Rigaku D/MAX-2400 diffractometer by using Cu Kα radiation (100 mA, 40 kV). The crystallite size (d) of the catalyst is calculated from the Scherrer Eq. (1).

$$d = \frac{0.89\lambda}{\beta \cos\theta} \qquad (1)$$

Where d = the mean diameter of crystallite, the Scherrer constant = 0.89, the X-ray wave length ($\lambda = 1.54056$ Å).

The Fourier transforms infrared spectroscopy (FTIR) used for the measurement of the prepared catalyst was recorded in the range of 400-4000 cm^{-1} on Shimadzu 8400 Fourier transform infrared spectroscopy (FTIR) spectrometer**.** Scanning electron micrographs (SEM) and SEM-EDX were recorded on Zeiss EVO 18 instrument. The accelerating voltage was used 15 kV and magnification of 1000X was applied. The specific surface area of the catalyst was measured by using micromeritics ASAP 2020 analyzer by physical adsorption of N_2 at the temperature of liquid nitrogen (-196°C) with a standard pressure range of 0.05-0.30 P/P_o. The X-ray photoelectron spectroscopy (XPS) was used to monitor the surface composition and chemical states of the constituent element. The instrument used for the XPS analysis is Amicus spectrometer. For typical analysis, the source was operated at a voltage of 15kV and the current of 12 mA. The binding energy of the element was calibrated by the setting the main C1s line of adventitious impurities at 284.7 eV.

Catalytic activity measurement

The combustion catalytic tests for the oxidation of CO has performed under the following reaction conditions: The total weight of the catalyst is 100 mg with feed gas consisting of a lean mixture of 2.5 vol.% CO in the air and the total flow rate is maintained 60 mL/min, using a fixed bed tabular flow reactor. The air feed enter into the reactor was made free from CO_2 and moisture by passing through it CaO and KOH pellets drying towers. The reaction temperature increased from ambient to 250°C with a heating rate of 2°C/min. The catalytic experiments were carried out in steady state conditions. For controlling the heating temperature of the catalyst presence in the reactor controlled by a micro-processor based temperature controller. To monitor and control the flow rates of CO and air through the catalyst in presence of the reactor is done by digital gas flow meters. The oxidation of CO in presence of air over the catalyst can be represented by Eq.2.

$$2CO + O_2 \rightarrow 2CO_2 \qquad (2)$$

The gaseous products produced from the reactor after the reaction was analyzed by an on-line gas chromatograph (Nucon Series 5765) equipped with a FID detector, porapack q-column and methaniser for measuring the concentration of CO and CO_2.

RESULTS AND DISCUSSION

The result and discussion of catalysts characterization by following techniques and the activity of the catalyst for CO oxidation were given below.

Catalyst Characterization

The characterization of CoO_X catalyst reveals material structure analysis, pore size identification, phase identification and binding energy identification etc.

X-ray diffractogram of the catalysts

The X-ray diffraction (XRD) studies of the catalysts were carried out to identify the crystalline size and coordinate dimensions presence in the catalysts. The XRD pattern of the cobalt oxide catalysts produces the reactive calcination conditions display in Fig 1. The cobalt nitrate, cobalt acetate, and cobalt oxalate sample phase analysis is done by XRD studies. In the Cobalt nitrate precursor after RC condition, their diffraction peak at 2θ is 55.86 and corresponds to its lattice plane (4 2 2). The structure is face-centered cubic Co_3O_4 and crystallite size of the catalyst is 16.76 nm. In the Cobalt acetate precursor after RC conditions, their diffraction peak at 2θ is 34.19 with their corresponding lattice plane is (0 0 2) and its shown in figure.

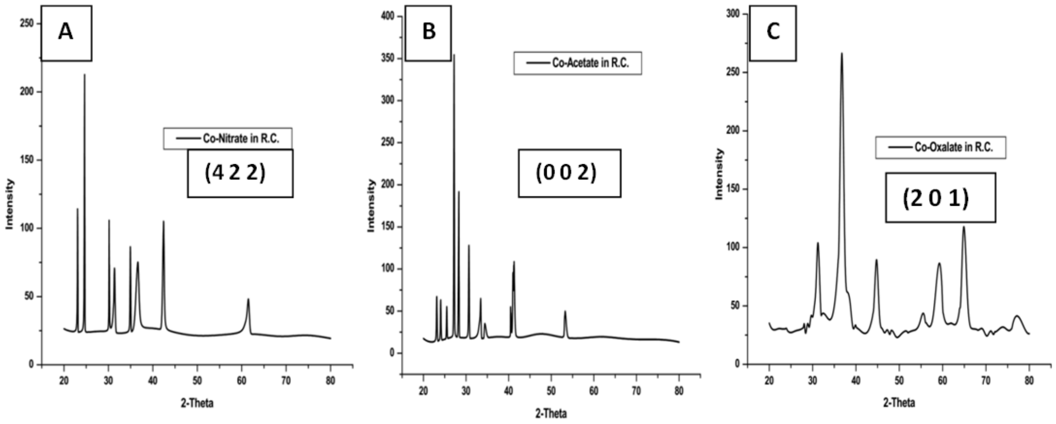

Fig. 1. XRD analysis of cobalt oxide catalyst prepared in RC conditions from the precursor of (A) Cobalt-Nitrate, (B) Cobalt Acetate and (C) Cobalt Oxalate

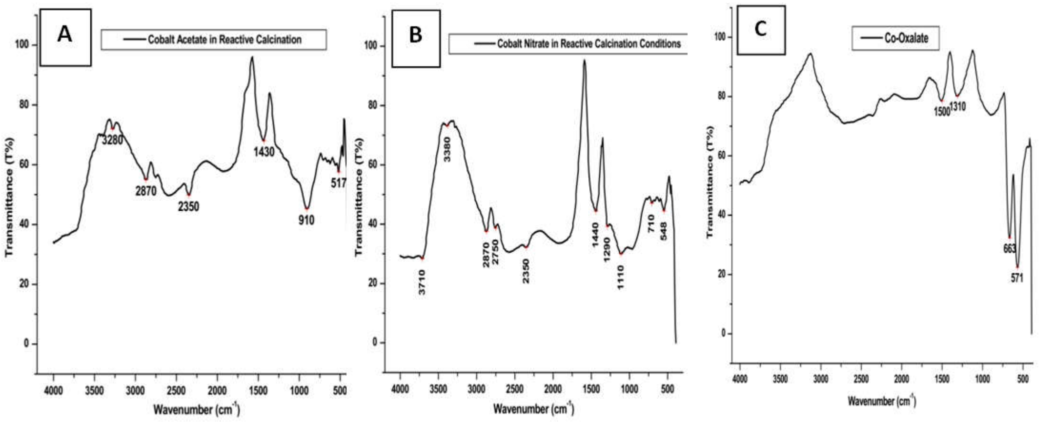

Fig. 2. FTIR spectra of cobalt oxide catalyst prepared in RC conditions from the precursor of (A) Cobalt Acetate, (B) Cobalt Nitrate and (C) Cobalt Oxalate

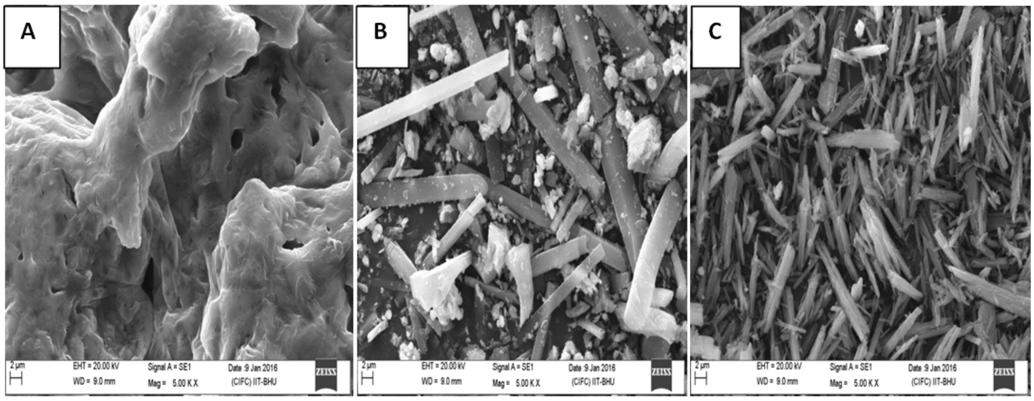

Fig 3. SEM image of CoOx catalyst produced by RC of a precursor of a) Cobalt Nitrate, b) Cobalt Acetate and c) Cobalt Oxalate

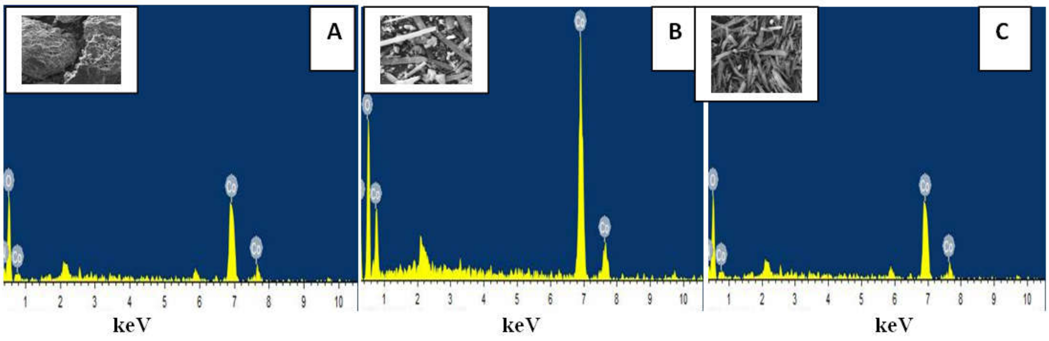

Fig 4. SEM-EDX image of CoOx catalyst produced by RC of a precursor of a) Cobalt Nitrate, b) Cobalt Acetate and c) Cobalt Oxalate

The structure is hexagonal and primitive CoO and the crystallite size of the catalyst is 34.19 nm. In the Cobalt oxalate precursor after RC conditions their diffraction peak at 2θ is 36.61 and corresponding its lattice plane is (2 0 1). The structure is hexagonal and primitive CoO and the crystallite size of the catalyst is 13.12 nm.

Fourier transforms infrared spectroscopy (FTIR)

The FTIR transmission spectra of the catalysts are shown in Fig. 2. In the invested region (4000-400cm^{-1}) to obtain the entire absorption spectra peaks to indicates the presence of different groups in all the three samples of the catalyst. The two strong bands at 1290cm^{-1} and 1440cm^{-1} in the cobalt nitrate sample indicates the presence of stretching vibrations of the metal-oxygen bond and also confirm the presence of Co_3O_4 spinel phases. The FTIR analysis of the CoOx sample from the precursor of cobalt nitrate in the transmittance conditions they are ten peaks we obtained. The IR band (3710 cm^{-1} and 3380 cm^{-1}) shows presence of O-H group, (663 cm^{-1} and 2870 cm^{-1}) shows presence of CoO_x group, 2750 cm^{-1} shows the presence of Cobalt Carbonyl group, (1290 cm^{-1}and 1440 cm^{-1} shows the presence of Co_3O_4), the weak band 1110 cm^{-1} shows the presence of COO group and (710 cm^{-1} and 548 cm^{-1}) shows presence of cobalt species respectively. In the Cobalt acetate sample, they are six peaks we obtained in the transmittance conditions. The IR band, 3280 cm^{-1} shows presence of O-H group, 2870 cm^{-1} shows presence of CoO group, 2350 cm^{-1}shows the presence of Physisorbed CO_2, 1430 cm^{-1} shows the presence of (C=O) group and (910 cm^{-1} and 517 cm^{-1}) shows the presence of cobalt species respectively. In the Cobalt oxalate sample, they are four peaks we obtained in the transmittance conditions. The IR band, 1310 cm^{-1} shows the presence of H-OH group, (663 cm^{-1} and 571 cm^{-1}) shows the presence of CoO group and the weak band 1500 cm^{-1} shows the presence of CO_3^{2-} group respectively. The intensities of FTIR peaks the presence of (CoO group) and (O-H group) impurities at (1430 cm^{-1} and 3380 cm^{-1}) decreases in the following order: Co-Nitrate > Co-Acetate > Co-Oxalate. The cobalt oxalate sample is highly pure as compared to Co-Nitrate and Co-Acetate sample.

Scanning Electron Microscopy Analysis

The SEM micrographs analysis the morphological of the cobalt catalyst sample. It represents clearly the microstructure of the CoOx catalyst samples in RC conditions. The SEM micrographs for various cobalt oxide catalyst samples granules prepared with different cobalt precursors show different granule sizes of 20-110 µm. As seen in the SEM micrograph, the particles size of catalyst were comprised, of course, fine and finest size grains resulted by RC of cobalt nitrate, cobalt acetate, and cobalt oxalate precursor respectively. The size of the particle is also confirmed from the crystallite size calculated based on XRD peak width and the particles of cobalt oxalate are highly porous, a smaller agglomerated, high surface area and uniform in nature compared to other two sample catalyst. The particle size of the catalyst produced by RC of cobalt Nitrate is very large and agglomerated than the catalyst produced by the RC of cobalt acetate and cobalt oxalate. It was also observed that cobalt was well distributed on the catalyst granule and different cobalt precursors were results in different crystallite size of cobalt oxide species in order of CoN > CoAc > CoO. After the activity tests, we get the results that the cobalt oxalate was high surface area and highly porous so that it oxidized more CO into CO_2 compared to other two catalysts.

Elemental analysis

It is clear from the results of SEM-EDX analysis that all the samples of catalyst were pure due to the presence of Co and O peaks only. The SEM was operated at 20kV and after the SEM micrographs were taken, the elemental mapping was performed to determine the elemental concentration distribution of the catalyst granules by using Isis 300 software. The cobalt oxide catalyst samples in fig. 4 were cut using an ultra microtome in order to perform SEM-EDX on different marks of cross-sectioned catalyst *granules* to determine the concentration of cobalt at different locations on the catalyst granular surfaces. Then, we observed that the high concentration of cobalt on the external surface of the cobalt oxalate sample. The SEM-EDX result confirms that the cobalt catalysts had their cobalt mostly located on the pores. The atomic abundance percentage of elements presence in the catalyst represent in Table 2. The cobalt surfaces reconstruction during the prolonged exposure to CO gas or aggregation of smaller nanoparticles into bigger size particles. The surface reconstruction behavior of a cobalt oxide metal surface such as cobalt metal has been observed in the presence of adsorbing CO gas. Table 2 displays the relative elements atomic (%) of Co and O species present in the surface layers of the catalysts sample. It is clear from the table the atomic percentage of Co is higher than O in cobalt acetate and cobalt oxalate precursor prepared the catalyst. The atomic percentage of cobalt in cobalt nitrate, cobalt acetate and cobalt oxalate in RC conditions is 28.89%, 58.87%, and 71.23% respectively. The atomic percentage of oxygen in cobalt nitrate, cobalt acetate and cobalt oxalate in RC conditions is 71.11%, 41.23%, and 28.77% respectively. The oxygen presence in the CoO precursor prepared catalyst is lower in compared to other two cobalt catalysts. It shows that there is oxygen deficiency in the cobalt oxalate (CoO) precursor prepared catalyst so that it creates the high density of active sites as a result enhancing the activity of the catalyst.

Table 2. The atomic abundance of elements measured over different catalysts

Catalyst	Elements atomic (%)	
	O	Co
CoN	71.11	28.89
CoAc	41.13	58.87
CoO	28.77	71.23

Table 3. The chemical state and binding energy of cobalt oxide catalysts in XPS analysis

Sample	Chemical state of Elements		Binding energy of Elements	
	Co	O	Co	O
CoN	Co_3O_4	Organic C=O	782.796	532.806
CoAc	CoO	Organic C-O	782.64	532.274
CoO	CoO	Organic C-O	782.79	532.148

XPS analysis

With the help of XPS analysis, we can get the surface valence state, binding energy and chemical state of cobalt oxide catalyst. In Table 3, we display the XPS spectra in the Co_2p and O1s regions.

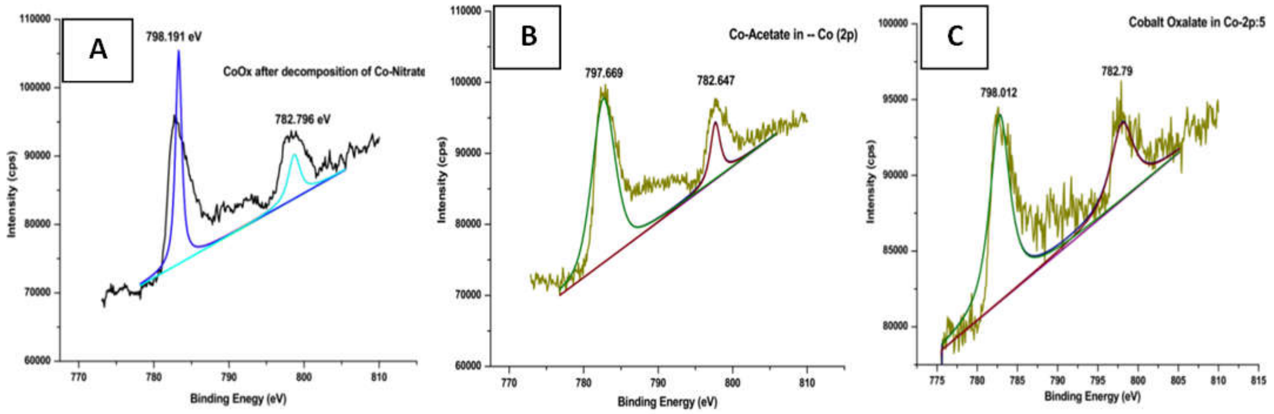

Fig.5. XPS analysis of Co(2p$_{3/2}$) in CoOx catalyst produced by RC of a precursor of a) Cobalt Nitrate, b) Cobalt Acetate and c) Cobalt Oxalate

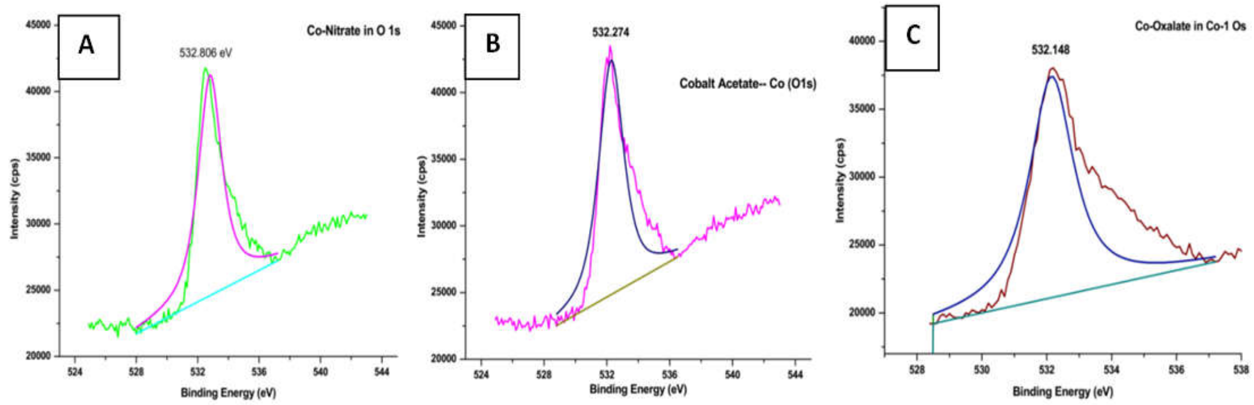

Fig.6. XPS analysis of O(1s) in CoOx catalyst produced by RC of a precursor of a) Cobalt Nitrate, b) Cobalt Acetate and c) Cobalt Oxalate

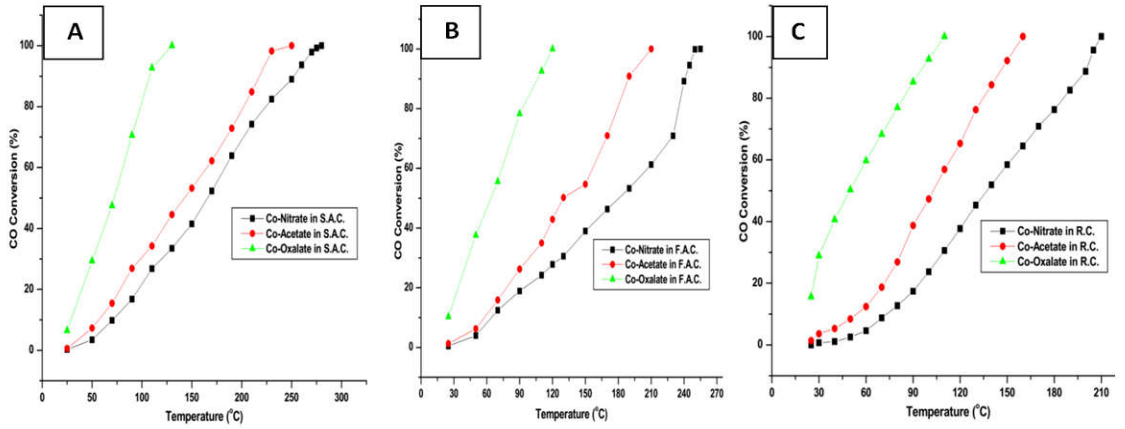

Fig. 7 The catalytic activity for CO oxidation by CoN, CoAc and CoO precursors in different calcinations conditions at (A) SAC, (B) FAC and (C) RC.

Table 4. The surface area, pore volume and pore size of CoOx catalyst from various Cobalt precursor in RC conditions

Catalyst	Surface Area (m^2/g)	Pore Volume (cm^3/g)	Ave. Pore Size (Å)
CoN	44.60	0.310	25.60
CoAc	71.59	0.430	35.60
CoO	93.402	0.590	48.70

Table 5. Light-off temperature characteristics of different CoOx catalysts for CO oxidation in different calcination conditions

Calcination Condition	Co-Nitrate catalyst			Co-Acetate Catalyst			Co-Oxalate Catalyst		
	T_i	T_{50}	T_{100}	T_i	T_{50}	T_{100}	T_i	T_{50}	T_{100}
RC	25ºC	150ºC	215ºC	25ºC	110ºC	165ºC	25ºC	50ºC	110ºC
FAC	30ºC	155ºC	255ºC	25ºC	125ºC	195ºC	25ºC	60ºC	115ºC
SAC	30ºC	165ºC	280ºC	30ºC	150ºC	230ºC	30ºC	75ºC	130ºC

The higher binding energy is preferably for the oxidation reaction. The Fig 5 and 6 displayed the spectra in the Co(2p) and O(1s) regions. In Fig 5, display the Co2p XPS spectra obtained from the different types of cobalt precursors after RC conditions. In the table, we analysis the Co ions in the CoN precursor is a Co_3O_4 phase and the Co ions in the (CoAc and CoO) is the octahedral symmetry as CoO form. The full-width half maxima (FWHM) of the $Co2p_{3/2}$ peak was found to be higher (4.3 eV) in the octahedral form. The binding energy of Co(2p) in CoOx catalyst at cobalt nitrate precursor, cobalt acetate precursor, and cobalt oxalate precursor is 782.796eV, 782.64eV and 782.79eV respectively. From the figure, it is clear that the binding energy of Co(2p) in cobalt oxalate precursor prepared catalyst is highest in RC conditions compared to other two catalysts. The binding energy of the Co2p transitions of the catalyst prepared form (cobalt nitrate and cobalt acetate) with low cobalt loading so that low reducibility of the cobalt oxide phases present in this catalyst. The prominent peak of the $Co2p_{3/2}$ level is deconvoluted into two peaks centered at 798.012eV and 782.79 eV presences respectively. Although, it can be proposed that the high binding energy is preferably for the CO oxidation reactions and its match with the results and discussions. The binding energy of O(1s) is displayed in fig 6. In general, there are two diverse types of oxygen in the catalysts with the binding energy 529.2–530 and 531.3–532.2 electron volt (eV) which could be recognized as chemisorbed oxygen (denoted as O_a, such as O_2^{2-}, O^-, OH^-, CO_3^{2-}, etc.) and lattice oxygen (denoted as O_l, such as O^{2-}), respectively. In our present study, the oxygen with a binding energy of 532.42 eV was the main form and could be assigned to chemisorbed oxygen (O_a). It is well known that the high amount of surface chemisorbed oxygen (most active oxygen) is preferable for enhancing the catalytic activity of oxidation reactions. The one carbon monoxide molecules adsorb on one cobalt sites, therefore the bridged bond accounted for highest in cobalt oxalate precursor prepared a catalyst.

BET surface area analysis

The surface area of different cobalt precursors like cobalt nitrate, cobalt acetate, and cobalt oxalate after RC is 44.60m^2/g, 71.590m^2/g and 93.402m^2/g respectively. From the Table 4, it is clear that the pore volume and pore size of cobalt oxalate precursor prepared catalyst is larger than other two catalyst samples. It will be noted that the specific surface area of cobalt oxalate precursor in RC conditions prepared catalyst is more than twice of that cobalt nitrate precursor prepared the catalyst. Clearly, the textural properties of cobalt oxalate precursor prepared catalyst are superior to active for CO conversion at low temperature. The average pore of cobalt oxalate precursor prepared catalyst 48.70nm which is slightly higher than other two catalysts. The larger more pores mean a number of CO molecules interacts with each other, and they show the better catalytic activity. The specific surface area of three CoOx catalysts measured by BET method and it match with the SEM and XRD results. The increases in pore volume and pore size of the catalyst by addition of Cobalt, so that the possibility of some pores collapsing during catalyst treatment after Co species addition (Khangale *et al*., 2014). The cobalt oxide catalyst surface area and pore size are most active for CO oxidation at low temperature, but it is several times deactivated by the trace amount of moisture present in the catalyst and under the dry condition the moisture content in the catalyst below 1ppm.

Catalyst performance and Activity

The cobalt oxide catalysts activity tests were carried out to assess the effectiveness of three different catalysts (CoN, CoAc, CoO) as a function of temperature for the oxidation of CO. The activity test was done in different calcinations condition like reactive calcinations (RC), flowing air calcinations (FAC) and stagnant air calcinations (SAC) conditions into the laboratory. The light-off characteristics are used to check the activity of the catalysts which is given in Table 5. The characteristic temperature T_i, $T_{50,}$ and T_{100} represents the initiation of the oxidation, 50% conversion and 100% conversion of CO. From the table and figure, we can get the result that the higher temperature was necessary for CO oxidation over all the three catalysts. The oxidation of CO was initiated at 30^{o}C, 30^{o}C and 25^{o}C over CoN, CoAc, and CoO respectively in RC conditions. The total oxidation temperature of CO is 110^{o}C for CoO catalyst, which was less by 55^{o}C and 105^{o}C than that of CoAc and CoN catalyst respectively in RC conditions. The order of activity for CO oxidation by various CoOx catalysts in different calcinations conditions follow this order RC > FAC > SAC. Therefore, the activity order for individual CoOx catalyst for CO oxidation is in accordance with characterization by XRD, SEM-EDX, FTIR, XPS, and BET as follows: CoO > CoAc > CoN. The improved Cobalt oxalate (CoO) precursor in RC conditions can be attributed to the unique structural and textural characteristics as the lowest crystalline size, highly isolated and the highest surface area which could be exposed a number of active sites presence in the catalyst it helps in the catalytic oxidation process. The higher amounts of textured pores presence in the catalyst which will be indicated for the adsorption of the reactants and desorption of the products. The higher amounts of adsorption make it easy for the oxidation process. The presence of partially reduced phase provides an oxygen deficient defective structure which creates the high density of active sites as a result of RC conditions. The cobalt surface oxidation behavior during a prolonged exposure to CO gas into smaller size particles to bigger sized particles of different cobalt precursors are studied in this paper. The surface restoration behavior of a metal surface such as cobalt metal has been observed in the presence of adsorbing CO gas. In the studies, we can also get that even small changes in lattice constant can significantly affect the binding energy of surface species. The other factors affect the reaction rates are a total number of the active sites which will depend on the exposed facets, the corner atoms, and the step edges present on the surface of the catalyst. The experimental result follows with a two-step reaction mechanism in which CO first reacts on the surface to obtain the OCO intermediate followed by dissociation to form CO_2 in the gas phases. To develops the different cobalt oxide catalyst by different calcinations conditions to developments of new generation catalysts.

Conclusion

The CO oxidation over the different types CoOx catalyst is significantly influenced by the crystallite size, shape, surface area, pore size of the catalyst particles. The activation energies for CO oxidation increase with the cobalt oxide catalyst particle size and it is proved that the smaller size particles are more active for CO oxidation. It can be concluded that the different types of calcinations strategy of the different types of cobalt precursor like CoN, CoAc and CoO has great influence on the activity of the resulting catalyst. The RC is one of the most appropriated calcinations strategies for the creation of highly active CoOx catalyst for CO oxidation. The performance of the catalyst in different calcination conditions for CO oxidation is as follows: reactive calcinations > flowing air > stagnant air and the order of activity to the individual CoOx catalyst for CO oxidation is as follows CoO > CoAc > CoN. The performance of the catalysts is in accordance with the characterization, therefore, it is suggested that the oxygen-deficient defective structure which produces high surface area of active sites. The catalytic activity of the CoOx catalyst relates to its unique structure, in which the bridge oxygen, between two Co atoms, is responsible for the oxidation reactions.

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