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Research Article

Comparative Study of Various Preparation Methods of CuO–CeO_2 Catalysts for Oxidation of n–Hexane and iso–Octane

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Abstract

The complete oxidation of n-Hexane and iso-Octane was studied individually in a fixed bed tubular flow reactor over CuO-CeO_2 catalysts synthesized via four different methods namely urea-nitrate combustion method, urea gelation/co-precipitation method, citric acid sol-gel method and co-impregnation method. Laser diffraction was employed in catalysts characterization. The results obtained from the complete conversion of n-Hexane and iso-Octane revealed that the CuO-CeO_2 catalysts prepared by urea-nitrate combustion method (UNC) showed the best performance than the catalysts prepared by other methods used in the present investigation. CuO-CeO_2 catalysts prepared by UNC method achieve total n-Hexane and iso-Octane conversion to CO_2 at lower temperatures of 280 ^{0}C and 340 ^{0}C respectively due to the larger surface area of the catalysts which increases the specific rate of reaction. © 2013 BCREC UNDIP. All rights reserved

Keywords: n-Hexane; iso-Octane; Copper-ceria; Urea-nitrate combustion; Catalytic oxidation

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1. Introduction

Hydrocarbons are considered as greatest contributor of the air pollution [1, 2] either directly as primary pollutant or indirectly as secondary pollutant [3] and harmful for their mutagenic and carcinogenic effects on human health [4,5]. Therefore proper destruction of hydrocarbons emitted from various stationary and mobile sources are required to prevent atmospheric pollution. Several techniques are available for the elimination of hydrocarbons such as adsorption [6], ozonation [7], thermal incineration [8], biodegradation [9], catalytic oxidation [10–14], etc. However, catalytic oxidation of hydrocarbon is considered as most developed cost-effective and efficient technology for the destruction of hydrocarbons. Various catalysts such as noble metals [15–19] and oxides of metal [20–23] are used for the destruction of hydrocarbons. However the disadvantages associated with noble metals i.e. the higher costs, the sensitivity to poisons and sintering at higher temperatures have spurred considerable efforts in search of low cost effective catalysts. CuO-CeO_2 catalysts have been reported to be one of such highest active catalysts. The lower costs, abundant availability and greater resistance to water, CO_2, and sulphur poisoning [24] are the advantages of CuO-CeO_2 catalysts over

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noble metal catalysts, which make them the strong contender for the catalytic oxidation.

The $CuO-CeO_2$ based catalysts can be grouped [25] into three categories: (a) CuO and CeO_2 catalysts [26, 27–31], (b) CuO catalysts doped with CeO_2 supported on alumina [32, 33–35], (c) CuO catalysts supported on mixed $Ce_xZr_{(1-X)}O_2$ oxides [36, 37]. It has been reported in the literature that the methods of preparation govern the properties of the catalysts [38].

The aim of the present investigation is to prepare $CuO-CeO_2$ catalysts with high activity at low temperatures for complete oxidation of lean hydrocarbon-air mixture experimentally and to screen out the best method of catalysts preparation. In the present work, n-Hexane and iso Octane have been used as typical representative of hydrocarbons and their combustion under lean conditions have been studied individually on catalysts prepared in the laboratory. This work utilizes the experience gained by earlier workers in catalysts preparation.

2. Materials and Methods

2.1. Preparation of Catalysts

Following four different methods were used for the preparation of copper-ceria catalysts:

2.1.1. Urea-nitrate combustion method

The catalysts were prepared by urea-nitrate combustion method as described elsewhere [26]. The metal nitrates and urea were dissolved in distilled water, under vigorous stirring at 80 ^{0}C for very short period. The gel obtained was placed in a preheated (500 ^{0}C) muffle furnace for 1h; the powders so obtained after auto-ignition were further calcined at 550 ^{0}C for 2 h.

2.1.2. Urea-gelation/ co-Precipitation method

Urea gelation/ Co-precipitation as described by Li et al., Fu et al. (a), and Fu et al. (b) [39–41] were used for the preparation of $CuO-CeO_2$ catalysts with Cu/(Cu+Ce) molar ratio equal to 0.15, using nitrate and urea salts as starting material. 50 ml doubled distilled water having specification: pH 7.1, conductivity 140 μm/cm were used for dissolving copper and cerium nitrate and urea as per the desired molar ratio. The solution was heated to 80 ^{0}C under vigorous stirring till the transparent gel was formed; the gel obtained was boiled at 100 ^{0}C for 1h. The precipitate was filtered and washed with de-ionized water; the solid received was dried at 80-100 ^{0}C for 1h. After crushing, the powder was calcined at 500 ^{0}C for 1h.

2.1.3. Citric acid Sol-gel method

$CuO-CeO_2$ catalysts were prepared via the citric acid sol- gel method as described elsewhere [42]. Ultrapure water having specification: pH 7.1, conductivity 140 μm/cm were used for dissolving copper and cerium nitrate according to the desired molar ratio. Citric acid as a chelating agent with a 1.3:1 ratio of the acid to metal ions was added, followed by the addition of polyglycol (10% weight of citric acid added), under continuous stirring with the help of Teflon coated magnetic stirrer with maintenance of temperature around 80 ^{0}C. The gel received was dried at 110 ^{0}C for 12h. The sample obtained were decomposed at 300 ^{0}C for 1h and finally calcined at 550 ^{0}C for 3 hours in a muffle furnace. The samples were further aged at 800 ^{0}C for 4 h.

2.1.4. Co-impregnation method

Co-impregnation method [43, 44] was employed to prepare catalysts using copper and cerium as a precursor. γ-alumina supports were used separately. Supports were impregnated with aqueous solution of copper nitrate followed by evaporation over steam bath with constant stirring. Then the catalysts were dried at 110 ^{0}C for 4 hours in an oven, and finally decomposed at

Table 1. The designation and elemental composition of the catalysts

S. No.	Catalysts	Methods of Preparation	Precursors
1	A	UNC	Nitrate
2	B	UGC	Nitrate
3	C	Sol-gel	Nitrate
4	D	Co-impgn.	Nitrate

Table 2. Particle size analysis of the catalysts

Catalysts	Prepn. Methods	Surface Area	Mean size of the samples (μm)	
		(m^2g^{-1})	Fresh Samples	Aged Samples
A	UNC	158	0.93	1.22
B	UGC	136	1.06	1.34
C	Sol-gel	143	0.98	1.23
D	Co-impgn.	125	1.02	1.38

500 ^{0}C in the muffle furnace in air. The designation and elemental composition of the catalysts are given in Table 1.

2.2. Catalysts Characterization

The particle size of the catalysts sample were analyzed by means of laser diffraction (Helium-Neon Laser, 5 Milliwatt) employed particle size analyzer (ANKERSMID, CIS-50, and U.S.A). Data for particle size are given in Table 2.

2.3. Experimental Set-up

The schematic diagram of the experimental set-up is shown in Figure 1. The experimental set-up used in the present study can be divided into following sections:

2.3.1. Feeding of hydrocarbon

The hydrocarbon was taken in an aspirator bottle (1) as shown in figure 1 which is connected to the sparger burette (2) with the help of silicon tubing. The hydrocarbon was fed in the burette by opening needle valve (3c) at a particular level in the burette. Then needle valve 3(c) was closed. The hydrocarbon vapour- air mixture was generated by sparging fine bubbles of secondary air through a fine epidermic needle connected at the bottom of the sparger burette as shown in the figure. After every drop in the level of the liquid by 0.1 ml in the sparger burette, the level of the hydrocarbon was maintained to its initial position by gentle opening the valve 3(c) and feeding the hydrocarbon from the aspirator bottle.

2.3.2. Feeding of gases

Air from a compressed cylinder was divided as primary and secondary air after purifying free of moisture and CO_2 by passing through a tower packed with CaO-KOH pellets. The secondary air having vapour of hydrocarbon mixes with the stream of primary air before entering into the reactor. A safety device in the form of Mercury sealing was provided in the feeding line to check any pressure building up as shown in the Figure 1

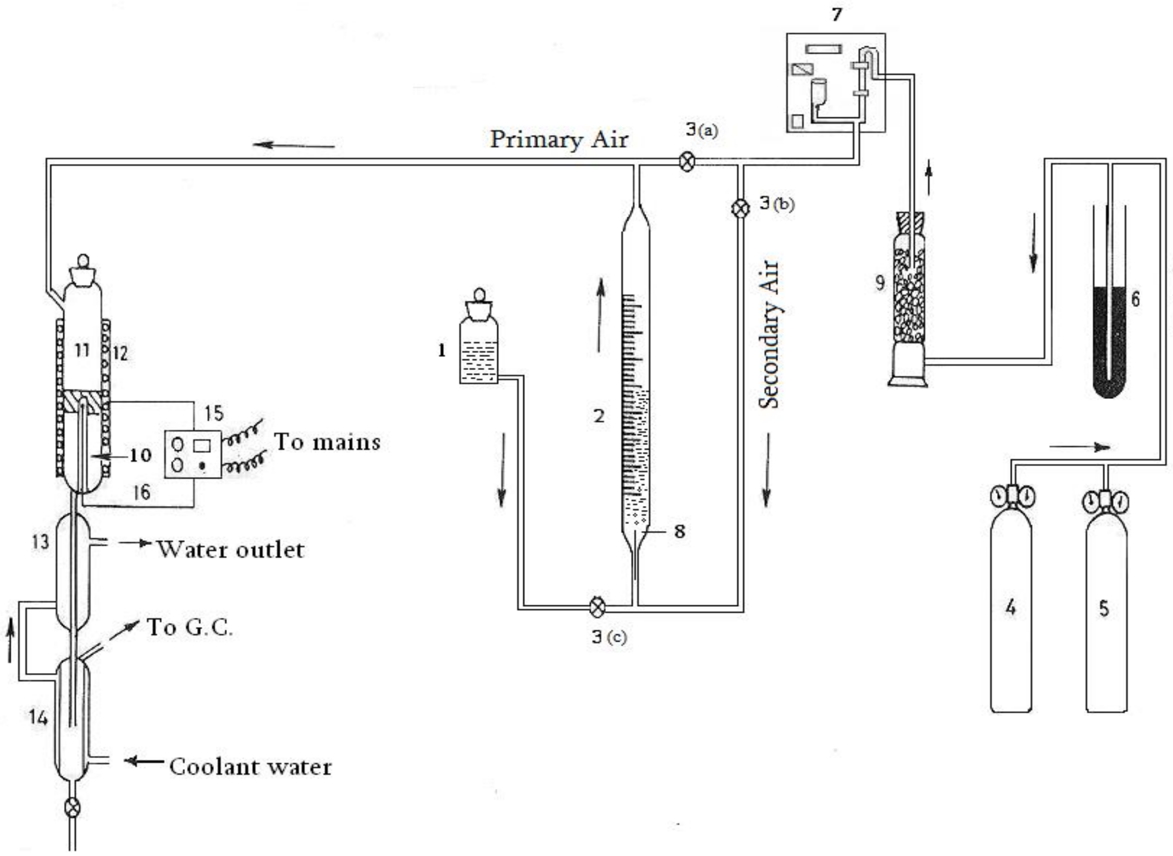

Figure 1. Schematic diagram of experimental set-up: 1. Aspirator bottle, 2. Sparger burette, 3. Needles valves, 4. Air cylinder, 5. Nitrogen cylinder, 6. Mercury seal, 7. Digital Gas flow meter, 8. Epidermic needle, 9. CaO+KOH tower, 10. Thermocouple well, 11. Reactor, 12. Split open furnace, 13. Condenser, 14. Reciever, 15. Temperature controller, 16. Thermocouple wire

2.3.3. Reaction system

100mg of the catalyst was weighed and diluted to 5ml volume by γ-Alumina. Then the catalyst was placed in the reactor over a bed of glass wool previously placed there.

2.3.4. Reactant and product analysis system

The reactant mixture and gaseous product after cooling to the ambient condition were analyzed by on-line gas chromatography.

2.4. Measurement of catalytic activity

The catalytic activity in the complete oxidation of hydrocarbon was evaluated in the fixed bed tubular flow reactor described by Prasad et al. 2009 [45]. About 100 mg of catalysts was placed in the reactor. The catalyst was heated at a constant rate of 5 °C/min, with the help of a microprocessor based temperature controller. The constant feed rate of total air (250 ml/min) was monitored with the help of a digital gas flow meter. The flow rate of secondary air in the sparger burette was controlled with the help of needle valve 3(b) such that 0.1 ml liquid hydrocarbon is vaporized in 15 minutes. The mixture of hydrocarbon vapour and air then enter the reactor. The n–Hexane/air and iso-Octane/air molar ratio of 0.004279 and 0.04166 respectively was maintained throughout the experiment. The temperature range varied between 50 °C to 450 °C. The reaction mixture was preheated in the pre-heater helical coil of the inbuilt reactor before entering the bed of the catalyst. Catalytic oxidation reaction takes place and the product stream after cooling through the condenser is sent to on line GC analysis equipped with FID. In the present work, the only products were CO_2 and H_2O and other byproducts were not found under most experimental conditions. The conversion of hydrocarbon was calculated as follows:

Considering the stoichiometric equation:

$$HC + Air_{(excess)} \rightarrow CO_2 + H_2O$$

Reading was recorded till the conversion becomes constant i.e. that there is no increase in conversion with further increase in temperature. At this condition hydrocarbon is totally oxidized to CO_2 and water vapour.

Therefore at temperature T_∞ conversion of hydrocarbon is X_A= 100%. T_∞ is the lowest temperature, above which hydrocarbon conversion becomes constant.

At intermediate temperature T, conversion of hydrocarbon is defined as:

$$X_A = \frac{F_{A0} - F_A}{F_{A0}}$$

where, F_{AO} is the molar flow rate of hydrocarbon in the feed. F_A is the molar flow rate of hydrocarbon in the product stream.

Now, $F_{AO} \propto A_{CO2}$ at T_∞, where A_{CO2} is the area of CO_2 chromatogram:

$$F_A \propto A_{CO2} \text{ at } T_\infty - A_{CO2} \text{ at } T$$

Therefore, $X_A = A_{CO2}$ at T

3. Results and Discussion

Various catalysts reported in table 1 have been evaluated for the oxidation of lean hydrocarbon–air mixture. The experimental procedure and product analysis are already discussed in experimental section. The $CuO–CeO_2$ catalysts activity synthesized by four different methods of preparation for oxidation of n–Hexane and iso–octane was evaluated. The conversion of respective hydrocarbon is calculated by knowing the inlet carbon loading and outlet concentration of carbon dioxide in the product stream since no CO was present in the product.

3.1. Activity of catalysts for n–Hexane oxidation

The complete conversion of n–Hexane to carbon–di–oxide as a function of temperature is shown in Figure 2. An inspection of the figure 2 shows that the catalysts (A) prepared by urea nitrate combustion method exhibit the highest activity among all catalysts achieving total conversion to CO_2 at 280 °C. Since the particle size of the catalysts (A) was smallest, catalysts (A) harbors largest surface area and hence showed the highest activity in comparison to the other studied catalysts. This is in accordance with the data presented in the Table 2. The development of repelling forces due to the presence of charges on the surface of catalysts inhibits the process of coagulation. This results in the uniform distribution of copper in the catalysts sample which is responsible for the highest activity of catalysts (A).

The lowest activity was shown by the catalysts (D) prepared by the co-impregnation method, achieving total conversion at a temperature of 390 °C, sintering of catalyst is the most likely factor responsible for lowest activity. Thus the activity order of catalysts preparation methods for n-Hexane oxidation is as follows: urea-nitrate combustion method > citric-acid sol gel method >

urea-gelation/ co-precipitation method > co-impregnation method.

3.2. Activity of catalysts for iso–Octane oxidation

The iso–octane conversion to CO_2 as a function of temperature is given in Figure 3. It is clear from the Figure 3 that the trend observed is very similar to the one found in case of n–Hexane oxidation with catalysts (A) prepared by urea nitrate combustion method being again the highest active catalysts achieving total conversion to CO_2 at 340 ^{0}C. In case of iso–octane oxidation the lowest activity was shown by the catalysts (B) prepared by the urea gelation co-precipitation method. The plausible explanation for this behavior of catalysts (B) might be the formation of bulk segregated CuO phase. Similarly the activity order of catalysts preparation methods for iso-Octane oxidation is as follows: urea-nitrate combustion method > citric-acid sol gel method > co-impregnation method > urea-gelation/ co-precipitation method.

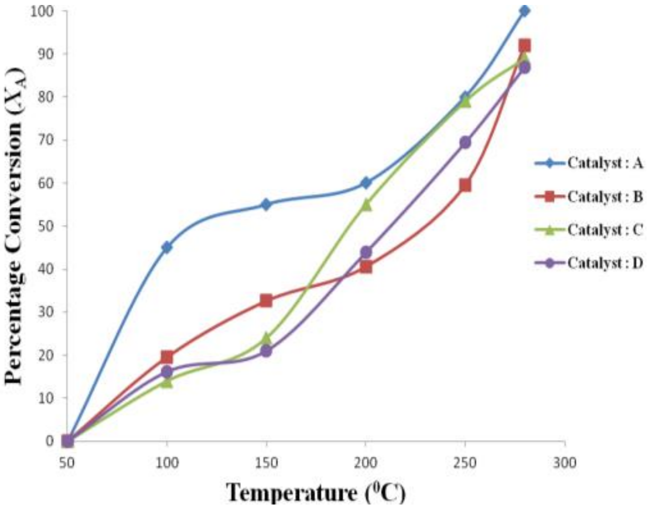

Figure 2. Percent conversion of n-Hexane at various temperatures with different catalyst.

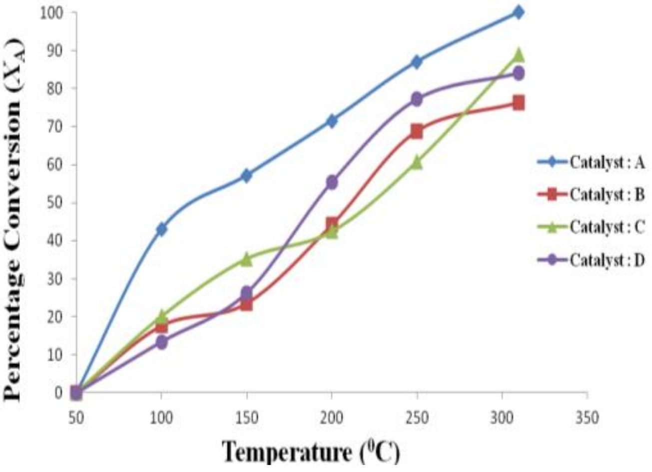

Figure 3. Percent conversion of iso-Octane at various temperatures with different catalysts.

A number of catalysts have been reported in the literature for oxidation of hydrocarbons viz. n-Hexane [10,13,46] and iso-Octane [47–50]. However, most of the studies in case of iso-Octane have been reported on its partial oxidation [47–50]. Hence cannot be directly compared with the present investigation.

4. Conclusions

The catalytic oxidation of n-Hexane and iso-Octane under lean conditions was carried out separately using $CuO-CeO_2$ catalysts prepared by four different methods. The results obtained from the complete conversion of n-Hexane and iso-Octane revealed that the $CuO-CeO_2$ catalysts prepared by urea-nitrate combustion method (UNC) showed the best performance than the catalysts prepared by other methods used in the present investigation. $CuO-CeO_2$ catalysts prepared by UNC method achieve total n-Hexane and iso-Octane conversion to CO_2 at lower temperatures of 280 ^{0}C and 340 ^{0}C respectively due to the larger surface area of the catalysts which increases the specific rate of reaction. Finally it was concluded that the methods of preparation of catalysts largely govern the activity of catalysts and urea-nitrate combustion method (UNC) is the best method of catalysts preparation among the four different methods studied.

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