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*Corresponding author

Kimam Silas, Department of Chemical Engineering, University of Maiduguri, Nigeria

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Research Article

Modification of Monolith for Simultaneous SO₂/NO_x Removal from Flue Gas

Kimam Silas^{1*}, Habiba D Mohammed¹, Haruna M Kefas² and Wadinda J Mamza¹

¹Department of Chemical Engineering, University of Maiduguri, Nigeria

²Department of Chemical Engineering, Modibbo Adama University, Nigeria

Abstract

The aggressive acid treatment creates a wealth of oxygen-containing functional groups (-OH, C=O, C-O) on the surface of an adsorbent and acts as binding sites for catalyst support therefore, in this study, the surface of monolith (ACM) was oxidized via the acid modification and high temperature activation. Various types of catalysts from the precursors of cerium nitrate (CeO₂/ACM), copper nitrate (CuO/ACM) and cobalt nitrate (Co₃O₄/ACM) using the deposition precipitation synthesis method. The catalyst activity tests were investigated in a fixed bed reactor by passing a stream of flue gas generated by burning coal. The breakthrough study of SO₂/NO_x and the experimental results showed that for CeO₂/ACM catalyst the adsorption capacity of SO₂ was 24.3 and 18.5 mg/g for NO_x and for CuO/ACM catalyst they were 27.0 and 32.8mg/g and for Co₃O₄/ACM catalyst, values obtained were 10.6 and 49.7mg/g. FTIR, BET and FESEM analysis were performed. This investigation depicts the development of highly efficient adsorbent for flue gas treatment.

Introduction

Acid gases such as sulfur (IV) oxides SO₂ and nitrogen oxides compound (NO_x) are mostly emitted as a result of fossil fuel combustion in power plants and many other anthropogenic sources causing harm to humans, environment and the ecosystem at large [1]. Therefore, gas cleaning and proper utilization of such harmful gases has continued to be a topic of research and various adsorbents such as activated carbon, zeolites, metal-organic frameworks, amines, and monolith are investigated [2]. Because monolith adsorbent is composed of high surface area, it is used in heterogeneous catalysis as a catalyst or active support component, it is also used as three-way catalysts (TWCs) in automotive exhaust gas purification, electrochemical reactors, electric swing adsorption (ESA) [3], gas-solid, and gas-liquid-solid applications [4]. The application of monolith in adsorption process is still limited but gaining attention by the day [2,5]. The interaction between a catalyst precursor (example, metal oxide) and the support (example, monolith) is very critical in the gas cleaning operation, metal oxide such as CuO are impregnated on adsorbents to fasten the reaction between the adsorbates and the adsorbent. The oxidation of supports surfaces results in a more hydrophilic surface structure with a large number of oxygen-containing surface groups such as phenolic, carbonyl, and carboxylic formation on the surface linking to internal pore and the surface area which increases the accessibility of the impregnating metal oxides [6,7]. According to literatures, monolith has low specific surface area of 0.02-0.5 m²/g [8,9], therefore, surface improvement is a priority and the acid oxidation is simple and convenient means.

In the industrial catalyst application such as catalytic wet oxidation and soot removal from diesel engine exhaust, CeO₂ is considered as the most important oxides of rare earth elements [5,10]. Sumathi et al. [11], impregnated peanut shell activated carbon with cerium and removed NO/SO₂ simultaneously at temperature of 300°C but reported low adsorption capacity for NO_x of 3.17 mg/g. Athappan et al. [12], prepared a cerium/ACF but investigated only the NO reduction efficiency. Zhigang et al. [13] reported the kinetic model of SCR of NO with NH₃ over CuO/γ-Al₂O₃/cordierite support. Many researchers [14-16] impregnate CuO on monolith but none of the study performed the simultaneous SO₂/NO_x removal. The Co₃O₄ spinel structure, have great influence on catalyst activity [17]. Assebban et al. [18] dispersed Co₃O₄ on cordierite and clay monolith using Pulsed-spray evaporation chemical vapor deposition (PSE-CVD) for the oxidation of unsaturated hydrocarbon C₂H₂ and C₃H₄. NO_x-assisted soot oxidation was demonstrated with Co₃O₄-CeO₂ mixed catalyst synthesis through the sol-gel method. None among these work tried the deposition precipitation (DP) synthesis technique to test the catalyst for simultaneous SO₂/NO_x removal. The main procedure for deposition precipitation method involved the dissolution of the metal precursor together with a precipitation agent (urea) and the support followed by heating [19,20]. DP is a good method of synthesizing a catalyst [19] and a means at which insoluble catalyst precursor are formed [5]. There are few studies in the literature that used the DP method example, Cheng et al. [21] impregnate Ni(NO₃)₂·6H₂O on monolith, Buratti et al. [22] prepare Ni/SiO₂ catalyst, and Bitter et al. [23] demonstrated the loading of Ni(NO₃)₂·6H₂O on carbon nanofibre. These studies only concentrated on nickel nitrate hexahydrate as the precursor, and to our knowledge, there is no study that is reported which catalyst activity test was performed with the use of real flue gas that is generated in the laboratory rather, simulated gases were used.

The SO₂-NO_x adsorption process (SNAP) is a technology which targets to remove SO₂ and NO_x in a single step from a flue gas. For the first time, this work has reported the oxidation, activation and the separate modification of monolith with and CeO₂, CuO and Co₃O₄ catalysts through the deposition precipitation synthesis method. The catalyst activity tests showed the performance of each catalyst in the breakthrough studies and by the simultaneous SO₂/NO_x removal from flue gas generated through burning coal in the laboratory. To further explain the changes occurrence on the catalyst, The FTIR spectra, BET surface area analysis and FESEM imaging were carried out.

Materials and Methods

Chemicals

The chemicals nitric acid (HNO₃, 65%), urea (CH₄N₂O), cerium nitrate hexahydrates (Ce(NO₃)₂·6H₂O) copper nitrate hexahydrates (Cu(NO₃)₂·6H₂O) and cobalt nitrate hexahydrates (Co(NO₃)₂·6H₂O) were purchased from Sigma-Aldrich, Malaysia. All the chemicals and reagents used were of analytical reagent (AR) grade. The ceramic monoliths were purchased from Beihai Huihuang Chemical Packing Co. Ltd., China.

Catalyst preparation

The method of acid (HNO₃) treatment for the oxidization of the monolith was used. The monolith was immerse completely into the acid for 24 hr, then the adsorbent was filtered and washed with deionized water several times and placed to dry in an oven at 70oC for 24 hr, similar procedure was reported by Liu et al. [15]. At the heating rate of 5oC min⁻¹ to 800oC and then further 4 hr of heat treatment under constant nitrogen gas flow (and cooling), the oxidized monolith was activated and the description of similar activation procedure was reported by Wang and Yonghui [24].

Deposition precipitation synthesis method

A three-neck closed 350mL reactor vessel equipped with pH meter, thermometer, and magnetic stirrer was loaded with 250mL deionized water, ACM and the metal precursor. The pH was adjusted to 3.5 by adding a few drops of HNO₃. After heating the mixture to 90oC at constant stirring of 300rpm, a solution of urea in 3mL of water was added. After 18 hr, the slurry was cooled to room temperature and filtered. The catalyst was thoroughly washed and dried at 120oC for 18 hr followed by gradual treatment under nitrogen flow at a heating rate of 3oC m⁻¹ to 500oC for 4 h.

The fixed-bed adsorption of SO₂/NO_x

The activity measurement of the activated carbon monolith catalyst was conducted in a fixed bed reactor. The mass flow meter was used to control the flow rate of the flue gas which was generated by burning coal in an electrical vertical furnace. Figure 1 shows the schematic diagram of the fixed-bed adsorption system. The column was operated in a down-flow mode and about 0.5g of the catalyst was charged into the reactor and preheated with N₂ at 100 mL/min and 120oC for 30 min to remove water vapor in accordance with the work of Zeng et al. [25]. The operating condition of the simultaneous SO₂/NO_x removal at atmospheric pressure was set at; 100oC of the column temperature and 400 mL/min of flue gas flow rate. A stream of flue gas was passed into the reactor while the concentrations of SO₂ and NO_x of the inlet and the outlet of the reactor were measured simultaneously by an on-line flue gas analyzer (T-350, Testo Company, Germany). The gas flow through the adsorption column was continuously monitored for an additional time after the exhaustion time. Each and every experimental run was repeated three times to increase the precision of the results and the average value was reported.

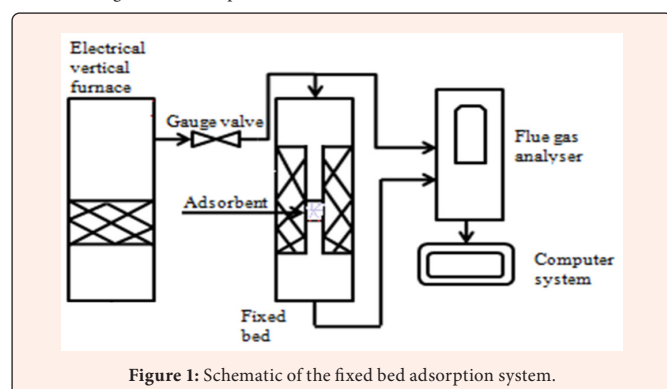


Figure 1: Schematic of the fixed bed adsorption system.

Characterization

The Fourier transform infrared (FTIR) spectroscopy which consisted of liquid nitrogen cooled mercury-cadmium-telluride detector and a spectra tech diffuse reflectance accessory (Thermo Nicolet AES0200682) was used. Samples of particle

size <45 lm were first dried for 24hr at a temperature of 383 K. The dried samples were mixed with finely divided KBr at a ratio of 1:100. FTIR spectrum was recorded at a resolution of 4 cm⁻¹ and with 200 scans per sample and an aperture setting of 15. A previously recorded background spectrum of water vapor was subtracted from the spectrum of each sample. The specific surface area and nitrogen adsorption-desorption data at 77 K were measured using Micrometrics ASAP2020 on powders previously outgassed at 200oC for 4 h under inert gas flow to remove water and other atmospheric contaminants; SBET was determined according to the Brunauer-Emmett-Teller (BET) method. The morphology of the adsorbent was observed through a field emission scanning electron microscope (FESEM, Nova NanoSEM 230, FEI, USA).

Results and Discussion

Adsorption capacity of SO₂/NO_x

The adsorption capacity (q) for SO₂ and NO_x were determined by numerical integration of the data using the breakthrough curve and the time equivalent to the total or stoichiometric capacity of the column and for a particular adsorbate, q was calculated using the following equation [26]:

$$t = \int_0^t (1 - \frac{C_t}{C_0}) dt \quad (1)$$

where t is the time equivalent to the total or stoichiometric capacity, C_t is the concentration of adsorbate at time t, and C₀ is the feed concentration of the adsorbate. The adsorption capacity was determined from eq. (2) [26]:

$$q = \frac{y \cdot F}{M} t \quad (2)$$

where y is the mole fraction of the adsorbate in the feed, F is the volumetric flow rate (mL/min) and M is the amount of the adsorbent (g). The breakthrough curve was constructed from the experimental data and the performance of each adsorbate was evaluated from the tb curves. Similar breakthrough studies can be found in the literature [27,28]. Figure 2 shows the breakthrough curve as the ratio of outlet concentration to the inlet concentration for both adsorbates (SO₂/NO_x) and for each adsorbent.

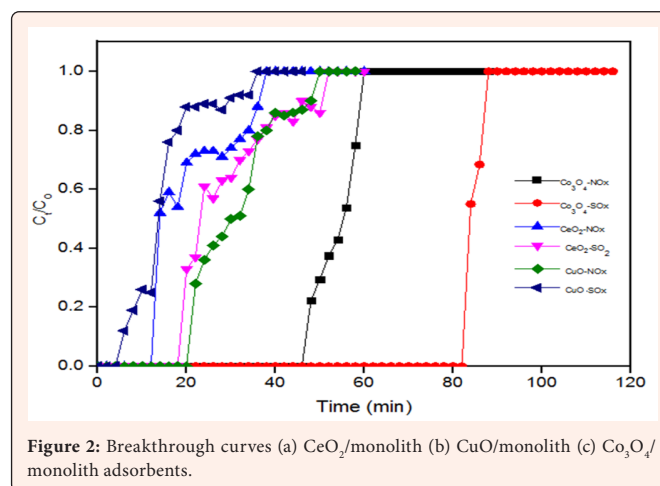


Figure 2: Breakthrough curves (a) CeO₂/monolith (b) CuO/monolith (c) Co₃O₄/monolith adsorbents.

From Figure 2 the adsorbent could adsorb SO₂ and NO_x successfully (100%) for certain period of time. According to the CeO₂/monolith performance, the breakthrough time occurs after 16min for both SO₂ and NO_x, this is an indication of the competition of the adsorbates on the active site and the adsorption affinity of the adsorbent. This also, implies that the adsorbates are all adsorbed on the surface in close proximity. The exhaustion time for the adsorbates differs and it was found that SO₂ adsorption was exhausted after 44min while for NO_x it was 32min.

The continuous passage of the adsorbate onto the catalyst leads to lesser amount adsorbed due to the reduction in the number of available catalyst active sites caused by the gradual SO₂/NO_x deposit in the pores. Therefore, the breakthrough time decreased and the exhaustion time increased with time. A study has revealed that cerium oxide has the potential for oxygen storage and redox property [29] and the enhanced oxidation of NO to NO₂ is a contributing factor for the removal of NO_x over this

catalyst. The synthesis method influences the catalysts textural properties and active site availability therefore, as a result it affects the performance of the catalytic activities. The breakthrough time for SO₂ and NO_x on the CuO/monolith adsorbent, were 6 and 20 min while the saturation times were 34 and 48 min respectively. The performance of the adsorbent to high NO_x affinity is a good breakthrough because in the literature, very low values were reported. Sumathi et al. [11] reported the low NO_x adsorption capacity of 3.17 mg/g. One of the reasons for the adsorbents high NO_x adsorption affinity is the concentration of the adsorbates which has almost equal proportion from the flue gas composition (±100-300 ppm SO₂ and NO_x).

In most of the work done with regard to simultaneous SO₂/NO_x removal, biased concentrations of the simulated flue gases were used ranging from 2000 ppm of SO₂ and 700ppm of NO_x with similar approach are available in the [30,31]. Other factors of important are the column temperature, flue gas stream's flowrate and most significantly the surface modification and catalyst impregnation, similar argument were [8,11]. Co₃O₄/monolith adsorbent depicts a better performance with SO₂ having the longest breakthrough time and exhaustion time compared to NO_x.

Functional groups result

FTIR is a tool that can characterize the functional groups on the catalyst [32]. Figure 3 show the FTIR spectra for the oxidized adsorbent.

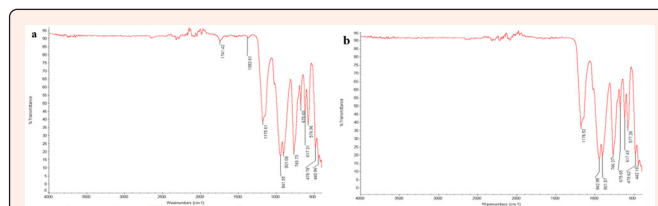


Figure 3: FTIR spectra for the (a) oxidized ACM (b) un-oxidized ACM.

FTIR spectra for the A weak peak of 1741.42 cm⁻¹ was observed for the oxidized ACM which is found to be within the range of the specific peak for the carboxylic acid as a result of HNO₃ oxidation. Absorption bands at 1741-1382 cm⁻¹ were changed after the acid modification, the range is characteristic of the presence of C-O- and N-O containing structures. The un-oxidized ACM showed a peak at 1176-943 cm⁻¹ which is less pronounced than oxidized ACM. Following the work of El-hendawy [33], the peaks at 941-440 cm⁻¹ and 942-442 cm⁻¹ for the oxidized and un-oxidized ACM respectively were considered as shoulder bands at lower wavelengths numbers, related to out-of-plane bending modes.

Table 1: BET surface area of the adsorbents.

Adsorbent	Break through time (min)		Exhausted time (min)		Ads.capacity (mg/g)		BET fresh catalyst		BET exhausted catalyst	
	SO ₂	NO _x	SO ₂	NO _x	SO ₂	NO _x	S.A.(m ² /g)	Vp(cm ³ /g)	S.A.(m ² /g)	Vp(cm ³ /g)
CeO ₂ /ACM	16	16	44	32	24.5	18.7	2.74	0.08	1.96	0.01
CuO/ACM	6	20	34	48	27	32.8	1.39	0.65	0.19	0.1
Co ₃ O ₄ /ACM	84	48	84	48	10.6	49.7	16.7	0.14	1.92	0.09

S.A= Surface area, Vp= Pore volume, Ads.= Adsorption

From Table 1, it can be deduced that there is surface area reduction due to the adsorbates deposit on the pores and according to the results, there were less active sites for adsorption to take place on the exhausted catalyst compared to the fresh catalyst. The surface area decreased significantly after catalyst activity and this observation indicates that the preparation method used in this study allows CeO₂, CuO, and Co₃O₄ particles to be located in the most internal part of the pores. Furthermore, there is a change in the structural parameters which demonstrated that the adsorbates were deposited on the surface and were considered to be the main reason for the catalyst deactivation. A similar study reported that the reduction in the surface area and active site blockage was caused by the adsorbate [32]. The BET surface result of the CeO₂, CuO, and Co₃O₄ ACM after the catalyst activity reduces with 28.5, 86.3 and 88.5% surface area reduction compared to the fresh ACM. The FESEM images of the ACM catalysts before and after the catalyst measurement are presented in Figure 4.

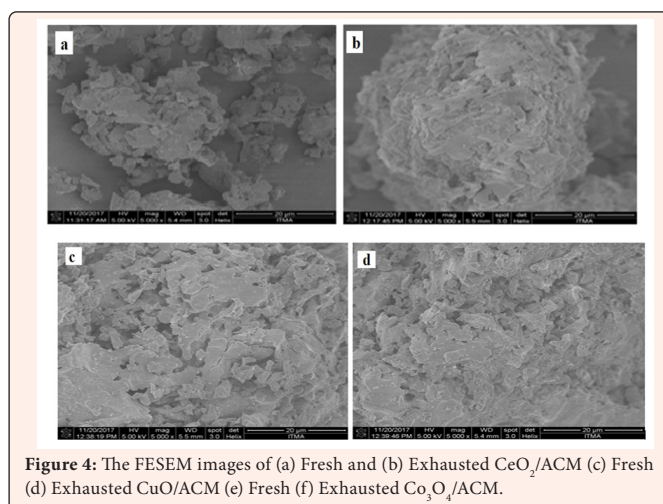


Figure 4: The FESEM images of (a) Fresh and (b) Exhausted CeO₂/ACM (c) Fresh (d) Exhausted CuO/ACM (e) Fresh (f) Exhausted Co₃O₄/ACM.

The FESEM analysis results shown in Figure 4 depicts that the adsorbent has tiny crystalline grains on the surface indicating the presence of dispersed cerium metal before and after the catalyst measurement. The exhausted catalyst displayed a hard-caked structure and was caused by the variable temperature during the adsorption process and the SO₂/NO_x deposit. In agreement to the above explanation, Jui et al. [34] reported that the adsorbent activity of a supported metal is related to the morphology that is the size and shape of the particle. The porosities observed on the fresh adsorbent were more abundant compared to the exhausted adsorbent as depicted. This is the indication of pore blockage as a result of adsorbate deposited on the surface area and in the pores of the also, the exhausted adsorbent displayed a caked surface. The change in morphology can be further seen in the reduction of surface area of the adsorbent after the adsorption process.

The richness of the Co₃O₄/monolith FESEM image is associated with the developed surface area and abundant active sites. The BET surface analysis proves that when compared to the other adsorbents, the Co₃O₄ precursor was dispersed appropriately on the monolith support with the help of oxygenated functional groups displayed by the FTIR spectrum. Figure showed that the influence of adsorption and heat on the adsorbent (since it stayed more than the other catalysts in operation according to breakthrough and exhaustion results) has resulted to the agglomeration of the adsorbent.

Conclusion

In conclusion, the breakthrough study of the simultaneous SO₂/NO_x removal from laboratory generated flue gas using the modified CeO₂/ACM, CuO/ACM and Co₃O₄/ACM catalysts were performed. The acid modification of ACM led to the fixation of weakly functional groups was shown by the FTIR spectra while the oxidation has extensively increased the amount of total acidity by surface oxide groups (carboxyl) with an intensity of 1741.42 cm⁻¹ absorption peak for the oxidized ACM. The CeO₂/ACM and the Co₃O₄/ACM catalysts affinity to SO₂ adsorption were higher compared to the NO_x while for CuO/ACM catalyst reverse was the case. The BET surface area of the fresh catalysts reduces considerably when compared to the exhausted catalysts due to the adsorbate deposited in the pores and the variable temperature, this fact resulted



to the hardening of the catalyst as the FESEM images depicted. It is expected that the simultaneous removal of the SO_2/NO_x gases from flue gas using a single process and at a low temperature of 100°C which is demonstrated in this work, will potentially change the economy and become a desirable option in the yarn towards the global mitigation of flue gas air pollution.

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