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Performance of Palm Shell Activated Carbon Impregnated with CeO_2 and V_2O_5 Catalyst in Simultaneous Removal of SO_2 and NO

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Abstract: This study examined the SO_2 and NO sorption capacity of modified Palm Shell Activated Carbon (PSAC). The PSAC was modified using vanadium and cerium metal oxides and the effects were studied for simultaneous removal of SO_2 and NO at temperatures of 100-300°C. The weight percentage of metal added and their calcination temperature were also analyzed. It was found that cerium based PSAC showed a higher sorption capacity compared to vanadium. Calcination procedure plays an important role in provoking the oxide formation from the impregnated metal nitrates. A 10% loading of Ce onto PSAC resulted in 121.7 mg g^{-1} of SO_2 and 3.5 mg g^{-1} of NO sorption capacity g^{-1} of Ce/PSAC at temperature 150°C compared to vanadium which shows a much lower sorption capacity. Lower temperatures are favorable for the SO_2 removal and higher temperature are favorable for NO reduction.

Key words: Palm shell, SO_2, NO, simultaneous, cerium, vanadium

INTRODUCTION

Emissions from combustion processes such as sulfur dioxide (SO_2) and Nitrogen Oxide (NO) has been causing many environmental hazards, particularly acid rain and ground layer ozone formation. Reducing these emissions simultaneously has been a target for many environmentalists. Among the famous methods and technologies, dry sorption is still considered to be one of the most attractive ways to treat waste gases containing SO_2 and NO. This method has the advantage of being simple with less equipment, lower capital and operating costs and limited secondary waste generation (Ishizuka *et al*., 2000; Lin *et al*., 2003). At present, there are only a limited number of reports on simultaneous removal of SO_2 and NO by dry method. Some of latest reports include: Ca-based sorbent (Nimmo *et al*., 2004; Pisupati and Bhalla, 2008; Dahlan, 2008), CuO based catalyst (Liu *et al*., 2009; Rodas-Grapain *et al*., 2005), pure activated carbon (Zhu *et al*., 2005; Qiang *et al*., 2005), activated carbon impregnated catalyst (Ma *et al*., 2008), Activated Carbon Fiber (ACF) (Mochida *et al*., 2000) and honeycomb catalyst (Wang *et al*., 2004; Yanli *et al*., 2004).

Activated carbon seems to be one of the most promising bases for simultaneous removal because the system can run at temperatures below 200°C and space velocities lower than 1000 h^{-1} (Nishijima *et al*., 1980). Any cheap material with high carbon content, low in in-organics can be used as a raw material for the production of activated carbon (Ioannidou and Zabaniotou, 2007). One of the latest types of activated carbon is activated carbon made from waste oil palm biomasses. Palm shell has been used to produce activated carbon and its product has been widely used for many applications in particularly to remove gaseous pollutant such as SO_2, CO_2 and H_2S (Guo and Lua, 2002a; Guo *et al*., 2007; Lua and Guo, 2001). Aroua *et al*. (2008) have reported the usage of carbon molecular sieve produced from palm shell to remove CO_2, O_2 and N_2. Up to the date, PSAC have not been reported on NO removal nor simultaneous removal of SO_2 and NO by other researchers.

Hence, the aim of this investigation is to study the simultaneous adsorption of SO_2 and NO from simulated flue gas on PSAC by varying: (1) type of metal support, (2) effect of calcination, (3) weight percentage of metal support and (4) effect of adsorber temperature. Vanadium and cerium were chosen as the metal catalysts. Productive results could promote the use of palm shell made activated carbon in desulphurization and denitrification processes simultaneously, since the calcium-based sorbents systems have shown some severe limitations.

MATERIALS AND METHODS

Preparation of activated carbon catalysts: An activated carbon prepared by physical activation with CO_2 gas from palm shell was used in this study. Details of the

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preparation method can be seen elsewhere (Sumathi *et al*., 2010). The prepared PSAC was then impregnated with metal nitrate of an appropriate concentration to obtain around 5 to 12 wt.% of metal content (10 mL of solution g^{-1} of PSAC) in order to study the effect of metal loading. In this study vanadium (VH_4NO_3) and cerium nitrate ($Ce(NO_3)_3 \bullet 6H_2O$) were used as the metal catalyst. Cerium nitrate was diluted in water whereas vanadium nitrate in oxalic acid. During the impregnation, the solutions were heated at 70°C while constantly stirred until the liquid was totally evaporated. Then the samples were dried in an oven at 110°C for a period of 12 h. Finally, the prepared samples were heat-treated at 500°C for 4 h in the presence of argon to get the calcined catalysts. PSAC impregnated with vanadium is denoted as V/PSAC whereas cerium is Ce/PSAC.

Activity test: The simultaneous removal activity of the prepared sorbent was carried out in a fixed bed adsorber. Details of the activity test rig and diagram can be seen elsewhere (Sumathi *et al*., 2009a). The metal impregnated PSAC sorbent (1.0 g) was placed on borosilicate glass wool (0.05 g) in the center of the adsorber. A stream of gaseous mixture representing the simulated flue gas, containing SO_2 (2000 ppm) (50%), NO (500 ppm) (11%), O_2 (10%), 15% of relative humidity and N_2 (29%) as the balance, was passed through the prepared sorbents. The feed flow through the adsorber was controlled at 150 mL min^{-1}. The inlet and outlet concentrations of SO_2 and NO were measured using a wall-mounted flue gas analyzer (IMR5000/400) before and after sorption activity. The concentrations of SO_2/NO were recorded continuously for every minute until it reaches the breakthrough point. The activity of the sorbent towards SO_2/NO was expressed by SO_2/NO sorption capacity, which is defined by the breakthrough curves (C/Co versus t). C/Co is a dimensionless factor, where C is the outlet concentration of SO_2/NO (ppm) from the adsorber, Co is the initial concentration of SO_2/NO (ppm) and t is the sorption time (min).

For temperature effect studies, the temperature of the adsorber was varied from 100 to 300°C and for space velocity from 14000 to 36000 h^{-1}. Each and every experimental run was repeated at least three times to increase the precision of the results and only the average value was reported throughout this study. The repeatability was found to be sufficiently high with relative error less than 5%.

Characterization: The surface area characterization of the catalysts was determinated by Brunauer-Emmett-Teller (BET) surface area. The characterization was done using N_2 adsorption and desorption isotherms measured at 77 K using Micromeritis (ASAP 2000 Series) volumetric gas adsorption instrument. It provides area comparisons for fresh and aged catalysts.

RESULTS AND DISCUSSION

Effect of calcination: First the PSAC was impregnated with metal nitrate of an appropriate concentration to obtain 5 wt.% of metal content (10 mL of solution g^{-1} of PSAC). The activity test was done accordingly at a temperature of 100°C. In this part of the study, PSAC acted as a support holding the metallic phase. The PSAC as support shows some advantages, for instance, high surface area and porosity compared to other supports. Furthermore the presence of oxygen surface groups indirectly affects the catalytic activity of the metals i.e., cerium and vanadium (Sumathi *et al*., 2009b). These oxygen complexes are the significant groups which act as an anchorage sites that interact with the metallic phase to improve the metal dispersion (Rodriguez-Reinoso, 1998).

The prepared impregnated PSAC was then tested on simultaneous removal of SO_2 and NO. Simultaneous removal of SO_2 and NO was successfully achieved with metal impregnated PSAC. It was found metal impregnated PSAC could remove NO successfully compared to PSAC without the addition of metal oxides.

Figure 1 and 2 show the ability of impregnated PSAC before and after calcination on simultaneous removal of

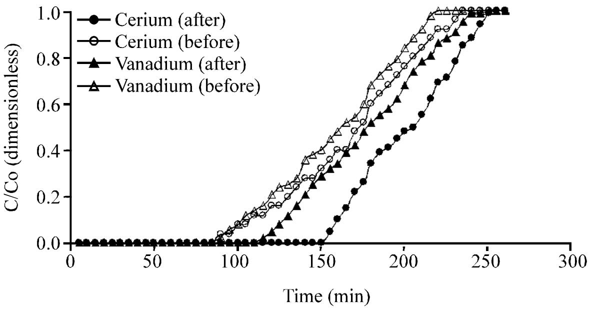

Fig. 1: SO_2 removal by Ce/PSAC and V/PSAC before and after calcination

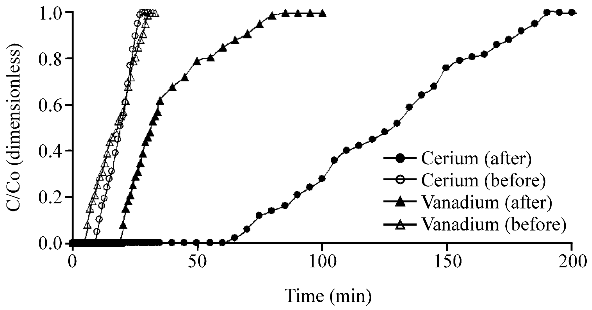

Fig. 2: NO removal by Ce/PSAC and V/PSAC before and after calcination

Table 1: Physical properties of metal impregnated PSAC (before and after calcination)

	Metal content (%)		Surface Area BET (m^2 g^{-1})		Pore volume (cc g^{-1})	
Samples	Before	After	Before	After	Before	After
PSAC	-	-	1250	-	0.399	-
Ce/PSAC	4.93	4.90	1071	1094	0.362	0.365
V/PSAC	4.87	4.90	1077	1096	0.360	0.362

Table 2: BET surface area and pore structure of the PSAC, V/PSAC and Ce/PSAC

Samples	PSAC		5	8	10	12
BET surface area (m^2 g^{-1})	1250.000	Ce	1094.00	962.00	854.00	807.00
		V	1100.00	954.00	867.00	812.00
Micropore volume (cm^3 g^{-1})	0.399	Ce	0.36	0.34	0.33	0.33
		V	0.36	0.34	0.33	0.32
Average pore diameter (Å)	16.000	Ce	15.50	15.30	15.30	15.00
		V	15.40	15.20	15.10	15.00

SO_2 and NO. With calcination show an increase in NO removal compared to without calcination. For SO_2 the change was very minor with calcination. This is because PSAC itself without the help of the metal oxides could remove SO_2; hence the removal of SO_2 was still good compared to NO. NO removal was poor if the samples were not calcined. This was due to the metal nitrates which interfere with the adsorption of NO.

During calcination, the impregnated support was heat-treated at a temperature high enough to provoke the oxide formation but sufficiently low to prevent unwanted sintering of the formed oxides (Marban and Antonio, 2001). This could be proven by analyzing the metal content before and after calcination. The amount of metal loaded on PSAC, before and after calcination is listed in Table 1. Results proved that no loss of metal occurred during the calcination process. Furthermore 500°C was selected for the temperature of calcination in order to preclude the burn-off of the activated carbon. Generally when a porous material is impregnated or doped with specified substances, pore blockage will happen. Hence, in this case, the fact that mineral crystallization and transformation of clay minerals to their semi-amorphous metaphases in char induced a pore blockage was extensively verified and confirmed by surface area analyses (Vassilev *et al*., 2002). The characterization of surface area and pore volume of the impregnated PSAC prepared with and without calcination is summarized in Table 1.

It was noticed that the surface area of Ce/PSAC and V/PSAC was lesser compared to the ones calcined. The evolution of pore volume was consistent with those of BET surface area, respectively. Similar type of results was reported by Zazo *et al*. (2009) for activated carbon impregnated with Ferum at different calcination temperature. Throughout calcination, the metal oxides were partially redistributed over the activated carbon with consequent loss in the microporosity. Calcinations also led to the opening and creation of new pores by the oxidation of the metals.

It was noticed that both Ce/PSAC and V/PSAC calcined samples showed higher NO reduction. This proves that calcined samples have higher solubility of the metal oxides into the pores of PSAC. This fact further improves the interaction between the support and the active phase, avoiding agglomeration and pore blocking (Lazaro *et al*., 2004). Ce/PSAC and V/PSAC with calcination resulted in an increase in porosity due to the opening of constructions in the char porosity and the development of new interconnecting pores by the metal oxides (Lazaro *et al*., 2006). From Table 1 it was clearly proved that calcined Ce/PSAC and V/PSAC have more porous structure. Generally, the higher the pore surface area of the activated carbon, the larger is its reduction capacity.

Effect of metal loading: The effect of metal loading was analyzed by varying the weight percentage of cerium and vanadium content on PSAC. The activity test was done accordingly at a temperature of 100°C. The BET surface areas, pore volumes and pore diameters of pure PSAC, V/PSAC and Ce/PSAC are summarized in Table 2. From Table 2 it is shown that the surface areas and the pore volumes of the pure PSAC are larger than the impregnated PSAC. The surface area apparently decreased as the content of cerium/vanadium increased in PSAC. The micropore volume also decreased accordingly. The average pore diameter in the impregnated sorbents was around 15.0 to 15.5 nm.

It is proven that the impregnation of cerium/vanadium onto PSAC, significantly affects the total surface area. These observations indicated that the preparation method used here allows CeO_2/V_2O_5 particles to be located in the most internal part of the pores, blocking the narrowest microporosity. Consequently, this fact provides a first evidence of high degree of cerium and vanadium dispersion on the PSAC support.

The effect of percentage of cerium/vanadium loading onto PSAC was analyzed. Table 3 shows the breakthrough time of SO_2 and NO removal obtained

Table 3: Sorption capacity of SO_2/NO by Ce/PSAC and V/PSAC with different wt.% percentage of metal loading

Properties	5	8	10	12
SO_2				
Breakthrough time (min)				
Ce	265.00	290.00	295.00	240.00
V	200.00	215.00	245.00	202.00
Sorption Capacity (g pollutant g^{-1} PSAC)				
Ce	104.10	113.10	115.80	94.20
V	78.50	84.40	96.20	79.30
NO				
Breakthrough time (min)				
Ce	175.00	225.00	275.00	170.00
V	115.00	145.00	200.00	160.00
Sorption Capacity (g pollutant g^{-1} PSAC)				
Ce	2.02	2.60	3.17	1.96
V	1.30	1.70	2.30	1.80

simultaneously at 100°C over Ce/PSAC and V/PSAC with different loadings. Impregnation of cerium/vanadium resulted in significant increase in NO removal. Both Ce/PSAC and V/PSAC could remove NO successfully in the presences of SO_2. Table 3 shows that the removal of NO and SO_2 by Ce/PSAC was higher compared to V/PSAC at all the weight percentage loading studied. A study by Davini (2001) for SO_2 shows almost a similar trend whereby PSAC-V was better compared PSAC-Ni and PSAC-Fe. Basically the nature and surface chemistry of each metal derivative plays a different role. Furthermore the difference could be also due to characteristic nature and the reaction mechanism of raw PSAC, PSAC sorbents and the reactivity of the metal oxides (Davini, 2001). Hence, in this case, cerium performed better than vanadium as a metal catalyst to remove NO and SO_2 simultaneously. Only small increases were noticed for SO_2 with increasing cerium/vanadium loading.

This was due to the competitive adsorption of NO on the cerium loaded PSAC. The best simultaneous removal of SO_2 and NO was achieved at 10 wt. % for Ce/PSAC and V/PSAC. The sorption capacity of SO_2 and NO at different percentage loading was calculated and tabulated in Table 3. However Ce/PSAC showed higher sorption capacity of SO_2 and NO (115.8 and 3.17 mg g^{-1}, respectively) at 10 wt.% compared to vanadium. The desulphurization and denitrifcation capacity was found to increase linearly from low to high weight percentage of loading for both metals except at 12 wt.%.

It is easily understood that the low activity of metal impregnated PSAC was due to the low dosage and coverage of cerium/vanadium on the PSAC surface at lower metal loading. Whereas for higher loading (12 wt.%) resulted in metal sintering. Metal sintering means aggregation of cerium/vanadium metal species which occurs heavier onto PSAC. These aggregations are hardly to be reduced and thus resulting in decreased activities. Similar trends were observed by Zhu *et al.* (1999, 2000) for coal based activated carbon impregnated with vanadium and copper for NO reduction.

These data proved that cerium plays a major role in the removal of NO from the simulated flue gas. It is well known that cerium and vanadium has a powerful oxidizing (redox conversion between Ce^{3+} and Ce^{4+} and V^{5+} and V^{3+}) and oxygen storage property which could bring new oxygen surface groups such as C-O, C = O and COOH on the surface of PSAC (Tian *et al.*, 2009; Kaspar *et al.*, 1999). These properties could enhance the oxidization of NO to NO_2 and SO_2 to SO_3 which indirectly contributed to the highest simultaneous removal of NO and SO_2. Furthermore cerium and vanadium could also develop the reduction of NO to N_2 which indirectly contributed to the highest breakthrough time of NO in this system.

Thus the reaction mechanism of SO_2 and NO simultaneous removal could be described by adsorption and reduction of cerium/vanadium metal in the presence of oxygen in three steps as deduced by many researches for other types of metal oxides, i.e., (1) chemisorption of SO_2 and NO on the catalyst surface; (2) transfer of oxygen from catalytic sites to the carbon reactive sites and (3) desorption of oxygen from the carbon surface (Zhu *et al.*, 2000; Illan-Gomez *et al.*, 1999).

Effect of adsorber temperature: Temperature has a significant role on the overall simultaneous removal of SO_2 and NO processes. In this study only 10 wt.% of cerium/vanadium impregnated PSAC was considered. The operating temperature of the simulated flue gas system was aimed to be low because flue gas temperatures at most of the industrial stack burners range from 120°C to 250°C. All this while throughout the study a temperature of 100°C was employed. In order to understand further the effect of temperature, higher temperature i.e., 150 to 300°C was tested on these sorbents.

Figure 3 and 4 shows the experimental breakthrough curves of SO_2 obtained under various temperature

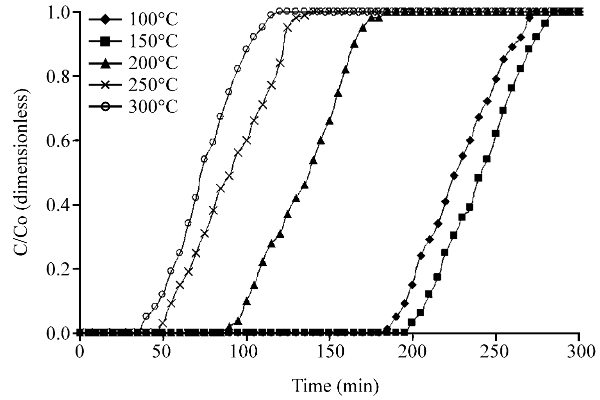

Fig. 3: SO_2 removal by Ce/PSAC at different operating temperature

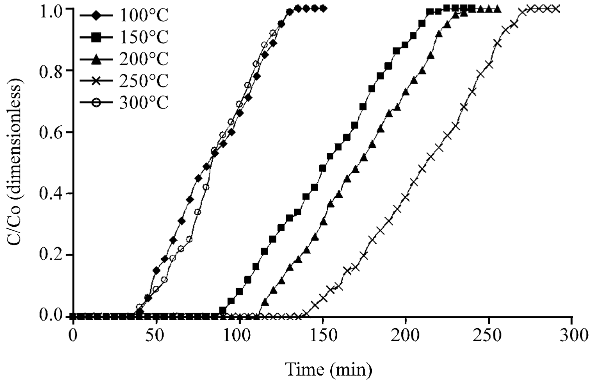

Fig. 5: SO_2 removal by V/PSAC at different operating temperature

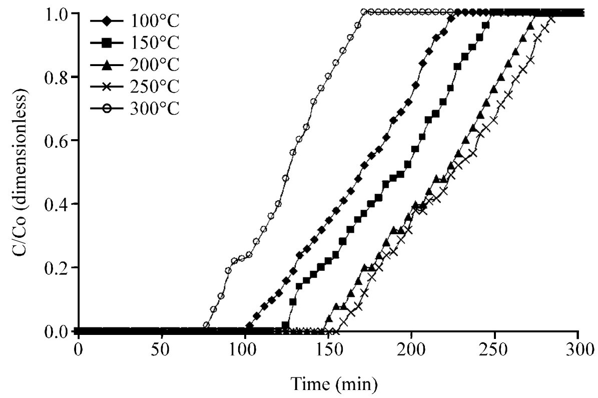

Fig. 4: NO removal by Ce/PSAC at different operating temperature

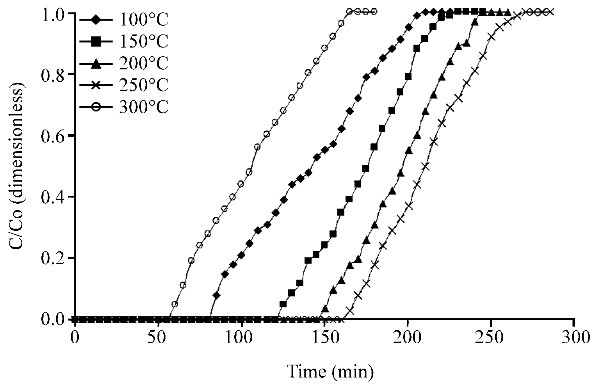

Fig. 6: NO removal by V/PSAC at different operating temperature

conditions over 10 wt.% of Ce/PSAC and V/PSAC. It was observed from Fig. 3 and 4, for SO_2 adsorption, as the operating temperature increased from 100 to 150°C, the breakthrough time of SO_2 increased from 285 to 295 min and 135 to 225 min for Ce/PSAC and V/PSAC, respectively. Zhang *et al.* (2006) reported that increased in operating temperature could enhance the chemical reaction rate and ionic diffusion rate of SO_2. Hence, in this case at temperature 150°C, SO_2 had higher ionic diffusion rate into the pores of impregnated PSAC.

For V/PSAC it was noticed that higher temperature i.e., 150 to 250°C promotes SO_2 removal and temperature 300°C deters the removal. However for Ce/PSAC, the removal deters as early as at temperature 200°C. The reported 100% removal time at 300°C was almost the same for both sorbents i.e., 35 min for Ce/PSAC and 40 min for V/PSAC. This could be due to kinetic energy of SO_2. It is known that at higher temperature SO_2 molecules loose their kinetic energies, thus making the adsorption exothermic process (Guo and Lua, 2002b). This condition indirectly lessens the amount of SO_2 adsorbed onto the pores of V/PSAC and Ce/PSAC.

Furthermore higher temperature would reduce the water accumulation and gas dissolving on the sorbent surface. Hence, the SO_2 sorption due to water molecules was reduced at temperature above.

On the contrary, in the case of NO as shown in Fig. 5 and 6, the sorption of NO was very favorable at higher temperature compared to the lower one for both adsorbents. At temperatures above 150 to 250°C the NO 100% removal time significantly increased from 140 to 180 min for Ce/PSAC and 120 to 160 min for V/PSAC. Mehandjiev *et al.* (1997) have suggested that at lower temperature (150-250°C) metal doped carbons show high catalytic activity due to the disassociation of NO chemisorption, accompanied by N_2O and N_2 evolution and oxygen accumulation on the catalyst surface. Besides that when the temperatures was low (<150°C) the impregnated cerium metal was most likely in a less active state, whereby at lower temperature, the capability of CeO_2 to

Table 4: Sorption capacity of SO_2 and NO by Ce/PSAC at different temperature

	Breakthrough time (min)		Sorption capacity (g pollutant g^{-1} sorbent)	
Temp. (°C)	SO_2	NO	SO_2	NO
Ce/PSAC				
100	285	275	115.8	3.2
150	295	300	121.7	3.5
200	195	330	76.6	3.8
250	160	345	62.8	4.0
300	130	210	51.1	2.4
V/PSAC				
100	145	220	56.9	2.5
150	235	240	92.3	2.8
200	250	255	98.2	2.9
250	285	280	111.9	3.2
300	160	135	62.8	1.6

store and release O_2 via the redox shift between Ce^{4+} and Ce^{3+} under oxidizing and reduction conditions, respectively was lesser (Qi and Yang, 2003) thus giving only a moderate removal of NO from the simulated flue gas.

Higher temperature shows a better removal of NO comparatively because Ce/PSAC would be in a more active state to oxidize and reduce NO. Moreover it is reported that at higher temperature activated carbon itself could decompose NO and reduce it to N_2 (Mehandjiev *et al*., 1996). Muniz *et al*. (1999) indicated that a catalytic reaction and chemisorption of NO on active centers must occur at temperature of more than 200°C. This was well proven by the reported analysis data in Fig. 5 and 6 and the sorption capacity reported in Table 4. Moreover higher temperature (>200°C) means less water accumulation on the sorbent surface and thus allowing the metal cerium or vanadium to become more active.

It has been also reported that the metal cerium/vanadium has high oxidation activity at high temperature conditions (Rodas-Grapain *et al*., 2005). A similar result was reported by Wang *et al*. (2004) using honeycomb activated carbon impregnated with vanadium oxide (V_2O_5/ACH). They found that SO_2 sorption decreased with increase in temperature and a stable sorption of NO at temperatures between 230 and 250°C. Ce/PSAC is playing a catalytic role alike vanadium at higher temperature by oxidizing and reducing NO to NO_2 and N_2.

However at temperature 300°C, it was detected that the removal of both SO_2 and NO starts deterring. This is owing to the oxygen-containing group formed on the surface of Ce/PSAC and V/PSAC, which is likely to attach to the carbon group PSAC to form CO and CO_2. This was proven when the analyzer starts detecting CO at this particular temperature. Additionally the cerium's oxygen group is expected to be attracted to the oxidized reactions of PSAC with oxygen. Serrano-Ruiz *et al*. (2008) studied and concluded that cerium dispersed on activated carbon aids the oxidation of the carbon surface with the onset of additional oxygen surface group retained in cerium. Consequently this process indirectly lowers the conversion of NO.

Table 4 shows the sorption capacity of Ce/PSAC and V/PSAC for SO_2 and NO at different temperature. At the temperatures reported, it was found Ce/PSAC gave higher sorption capacity of SO_2 and NO compared to V/PSAC at lower temperature. For SO_2, when the operating temperature was less than 150°C, the sorption capacity was high; whereas when the temperature was more than 150°C, the sorption capacity start reducing. Whereas for NO as temperature from 100 to 250°C the sorption capacity increase but then when the temperature was 300°C the sorption capacity reduced badly. Klose and Rincon (2007) reported that on a system of carbonaceous materials, simultaneous removal adsorption and reduction of NO occurs at temperatures lower than 300°C. At higher temperatures adsorption does not take place or it is too slow that it can be neglected. This could be one of the reasons for this observation.

A possible stoichiometric mechanism is represented as follows for Ce/PSAC and V/PSAC:

SO_2 sorption by cerium

$$xSO_2 + CeO_2 \Leftrightarrow xSO_3 + CeO_{2-x} \tag{1}$$

SO_2 sorption by vanadium

$$xSO_2 + V_2O_5 \Leftrightarrow xSO_3 + V_2O_{5-x} \tag{2}$$

NO reduction by cerium and vanadium

- CeO_2:

$$CeO_{2-x} + xNO \rightarrow CeO_2 + \tfrac{1}{2}xN_2 \tag{3}$$

- V_2O_5 through redox reaction becomes V_6O_{13}:

$$V_6O_{13} + 2NO \rightarrow 3V_2O_5 + N_2 \tag{4}$$

CONCLUSION

It can be concluded from the above observations and explanations that simultaneous removal of SO_2 and NOx from simulated flue gas can be done using PSAC impregnated with metal oxides. Ce/PSAC showed the best performance compared to vanadium impregnated PSAC at lower temperature. The highest number of sorption capacity i.e., 121.7 mg g^{-1} for SO_2 and 3.5 mg g^{-1} for NO was achieved by 10 wt.% of calcined Ce/PSAC at temperature 150°C. Ce/PSAC shows a catalytic effect towards the removal. Ce/PSAC could be a new and promising sorbent for simultaneous removal of SO_2 and NOx because not many research has been done using cerium oxide on activated carbon for this purpose.

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