The capacity of modified nickel catalysts derived from discharged catalyst of fertilizer plants for NO\textsubscript{x} treatment

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Abstract. In Vietnam for recent years, a large amount of hazardous waste containing nickel (Ni) derived from discharged catalyst of fertilizer plants has caused environmental problems in landfill overloading and the risk of soil or surface water sources pollution. Taking advantage of discharged catalyst, recycling Ni components and then synthesizing new catalysts apply for mono-nitrogen oxides (NO\textsubscript{x}) treatments is an approach to bring about both economic and environmental benefits.

This study was carried out with the main objective: Evaluate the performance of modified catalysts (using recovered Ni from the discharged RKS-2-7H catalyst of Phu My Fertilizer Plant) on NO\textsubscript{x} treatment. The catalysts was synthesized and modified with active phases consist of recovered Ni and commercial Barium oxide (BaO), Manganese dioxide (MnO\textsubscript{2}) / Cerium (IV) oxide (CeO\textsubscript{2}) on the support Aluminium oxide (\(\gamma\)-Al\textsubscript{2}O\textsubscript{3}). The results show that the modified catalysts with Ni, Ba, Ce was not more beneficial for NO\textsubscript{x} removal than which with Ni, Ba, Mn. 98% NO\textsubscript{x} removal at 350°C with the start temperature at 115°C and the \(T_{60}\) value at 307°C can be obtained with 10Ni10Ba10Mn/Al catalyst.

1. Introduction
Fertilizer is a small industry in Vietnam, whereas an average of 105,7 m\textsuperscript{3} of waste catalyst discharged every year from reforming, methanogenesis and NH\textsubscript{3} synthesis processes of one fertilizer plant. It becomes a source of solid waste pollution because of containing a lot of metal and toxic contaminants (Al\textsubscript{2}O\textsubscript{3}, ZnO, Fe\textsubscript{2}O\textsubscript{3}, Fe\textsubscript{3}O\textsubscript{4}, NiO, Cr\textsubscript{2}O\textsubscript{3}, CuO, CuCO\textsubscript{3}, CaO, CoO, MoO\textsubscript{3} and so on). In particular, the catalysts in the reforming and methanogenesis processes often contain Nickel (Ni) which is a heavy metal with the potential to pollute soil and water sources. Whereas, the Ni recycling from spent catalyst is a co-benefits approach including both environmental and economical solutions. According to Yang, the commercial Ni compounds cost is considerably high (21,46 USD/1kg in 2010 compared to 3,3 - 5,5 USD/1kg spent Ni) [1]. Regarding to the environmental application, Ni is known as a transition metal valuable which is emerging in term of research to be the precursor for polluted air treatment by catalysis, replaces for expensive precious metals [2-4]. Besides, in the field of industrial and civil polluted air treatment, using catalyst attached directly to the treatment device helps to transform unwanted substances into non-toxic form. In addition, mono-nitrogen oxides treatments (NO\textsubscript{x}, produced during combustion, included nitric oxide -NO and nitrogen dioxide - NO\textsubscript{2}) commonly exists in the emission of fossil-fuel or natural-gas-fuel processes which can lead to photochemical smog phenomenon. In regard with NO\textsubscript{x} emissions from internal combustion engines, three-way NO\textsubscript{x} decomposition (de-NO\textsubscript{x}) catalysts with the combination of several metal compounds had been commercialized. These types of catalyst based on precious metal precursors such as Pt (Platinum), W (Wolfram) or economic transition metals such as Ni, Ti (Titanium), Co (Cobalt), Ce (Cerium) and so forth. Acknowledge the situation, this research mainly questioned on the de-NO\textsubscript{x} performance of modified Ni catalysts derived from discharged catalyst of fertilizer plants.
2. Materials and methods

2.1. Components of the modified Ni catalysts

In the modified-catalyst preparation, Al$_2$O$_3$ is used as the catalyst support, recycled Ni solution from spent catalyst is used as the catalytic active sites along with Ba, Mn, Ce as catalytic supporting components. A proposition of Huong et al explain the mechanism of the NO$_x$ decomposition by reducing reaction on Ni sites could be as the following protocol: NO$_x$ molecules are adsorbed on Ni and decomposed into N$_2$; oxygen atoms separated from NO$_x$ is retained on the oxygen vacancies of catalyst, and then ionized and pumped out through the redox reactions [3]. In a number of related research [3-8], the de-NO$_x$ catalysts contain 0.1 – 30 wt% active phases by weight, whereas the Ni, Ce, Co active phase catalysts contain 3 – 20 wt%. Hence, this research prepared 5 – 30 wt% recycled-Ni/Al catalysts to test the NO$_x$ conversion catalytic capability of recovered-Ni components. Referring to the catalytic support, γ-Al$_2$O$_3$ is selected due to the high surface areas (about 130 - 280 m$^2$g$^{-1}$), uniform porous structure, greatly capable of dispersing active phases, extremely temperature resistant and very high mechanical strength [9]. Thus, γ-Al$_2$O$_3$ can increases catalytic durability in harsh reaction conditions [10]. Besides, be readily available and reasonably priced, it is no doubt that fine grain alumina is the most appropriate support for the high temperature NO$_x$ catalytic conversion.

Regarding to the Ba component in de-NO$_x$ catalysts which probably enhances catalytic reactions through types of mechanism as giving electrons for catalytically active metals promotion to a higher potential energy at common surface, which means they are less tightly bound [11]; releasing O$_2$ to form oxygen vacancies on the catalytic active phases; increasing catalyst defects on which NO easily adsorbs and dissociates [11]; increasing the NO$_x$ retention ability and time on catalyst surface [12]; basic sites play an important role in the NO$_2$ formation and adsorption - the intermediate products of NO decomposition to N$_2$ [13]. The amount of 5 – 20 wt% BaO are prepared on Ni/Al catalysts to investigate the NO$_x$ catalytic decomposition by basic Ba elements.

Be a representative of d-block transition metals, Mn component in de-NO$_x$ catalysts can generate anion-oxide defects which also improves the heat resistance for catalysts [14]. In the prepared catalyst, Mn could exist as Manganese dioxide (MnO$_2$ - black powders) and an inconsiderable amount of Manganese (III) oxide (Mn$_2$O$_3$) with the weight ratio is 5-20 wt% MnO$_2$. Besides, another type of transition metal is f-block elements with Ce as a significant component in de-NO$_x$ catalytic activity. It is reported that Ce catalytic performance in NO$_x$ treating reactions is only less favorable than alkaline and earth-alkaline metals. Studies by Zhu [15], Nguyen [6], Delmon [16], Pomonis [17, 18], Nitadori [19, 20] investigated on this component as favorable active or supporting sites in LaCoO$_3$, La-Sr-Ce-Fe-O, LaMnO$_3$ (M = Mn, Fe, Co) catalysts for the decomposition of NO$_x$. In de-NO$_x$ catalysts, Ce creates O$_2$ storages which reduces the "poisoned by oxygen" syndrome for active sites [15]; increase the metals dispersion on support thereby reduces the metal cluster problem and improves temperature resistance of supports [15]; demotes NH$_3$ product in NO$_x$ reduced reactions [6] (increases N$_2$ product in another words). Ce exists in the catalyst as the form of Cerium (IV) oxide (CeO$_2$ - light yellow crystals) and a small amount of Ce$_2$O$_3$, with the weight ratio is 5-20 CeO$_2$ wt%.

2.2. Experimental and analysis methods

2.2.1. Recovery Ni from discharged catalyst

The studied waste catalyst has the trade name RKS-2-7H which is used in the secondary reforming process at Phu My Fertilizer Plant. The Ni content in this waste catalyst is approximately 12.01%. The Ni recovery process from waste catalyst in laboratory scale is composed by three steps: material handling (crushed and sifted, then decoking); preparation (using 1.5M HNO$_3$ acid solution, 1:20 g/ml solid-liquid ratio, the temperature and time of digestion are 100°C in 1 hour) [21].

2.2.2. Catalyst preparation

Two main methods are used widely to prepare catalytic active phases are co-precipitation and metal impregnation. The co-precipitation method (precipitation deposition, Figure 1) is chosen due to the research orientation for making up to 40 wt% soaked-up metal components in catalysts. Nitrate
solutions are intentionally precipitated due to their good solubility in water, popularity and low-price (compared with oxalate or formate), easy removal after been furnaced (better than halides, sulfates). The pH value-stabilization at 10 is taken by NH₃ solution. At pH 10, the surface of alumina support will be negatively charged (so as the electrostatic interaction with Ni positively charged component could be better) [22]. The reducing pretreatment is performed to make up the metallic active phase (by the mixture of 30 vol% H₂ : 70 vol% N₂ at 550°C in 4 hours). For the 10Niₓ,10Ba10Mn/Al catalyst, pure Ni(NO₃)₂ 6H₂O is used instead of recovered Ni solution and there is no pH adjustment required.

Figure 1. Catalyst synthesis process from the recovered Ni

2.2.3. Model-based testing
After synthesis and pretreatment processes, catalysts would be tested for catalytic activity in NOₓ conversion through the micro-flow control system. Each catalyst loading is 0.1 g. The testing temperature is within range from the room temperature to 500°C. Each of CO/CO₂ and NO/NOₓ concentrations will be electrical recorded every 30 seconds by the Ulтратmat 6 and CLD 700 AL equipment, then expressed as graphs by the Virtual Bench program.

The testing air mixture includes: 500 ppm NO, 10000 ppm CO as a co-existing reducing agent, and N₂ as carrier gas. The gas volume flow rate is 10 L.h⁻¹ (~166 mL.min⁻¹). The gas hourly space velocity value (GHSV) is 110000 hours⁻¹.

2.2.4. Physicochemical properties analyzing methods
The specific surface area of samples are measured – including the pore size distribution by the BET (liquid nitrogen adsorption method). Chemical properties of catalysts are determined by the XRD
method (X-ray diffraction) in regard to existing crystal phases. The BET and XRD instrument are TriStar 3020 and Bruker AXS D8 respectively.

2.2.5. Data processing and analysis

The original data of gaseous concentrations are demonstrated as light-off graphs (conversion efficiency vs temperature) to identify the specific values: $T_{60}$, the conversion beginning temperature, the maximum conversion efficiency in the tested temperature range, the conversion efficiency at 350°C. Here are the much detail definition:

$T_{60}$: the temperature at which the conversion efficiency is 60%. For the NO concentration input is 500 ppm (613 mg/Nm$^3$ NO), 60% conversion efficiency is appropriate to adapt local environmental standards (250 mg/Nm$^3$).

Conversion efficiency at 350°C: according to related research showing the peak performance of NO$_x$ at about 350°C [4, 7, 8]. This value can express the low-temperature de-NO$_x$ performance of catalysts as well as be comparative to other related studies.

3. Results and discussion

3.1. Effect of the Ni active sites to catalytic activity

The tested Ni content changed from 5% to 10%, 20% and 30% by weight and denoted as xNi/Al (x = 5, 10, 20, 30 respectively). It can be seen that when Ni content increases from 5% to 10% by weight, the catalytic activity increased significantly (16% up to 63% at 350°C). However, it is not favorable for NO$_x$ conversion as increasing the Ni content up to 20 or 30 wt%.

Results by XRD of the Ni/Al catalyst show that the Ni peaks appear at positions of $2\theta$ = 45° and 52°, which indicates the metallic Ni existing on the surface (Figure 2). When Ni content in the samples gradually increase, the intensity of Ni° peaks also gradually increase along with a decrease in the intensity of the Al$_2$O$_3$ peaks. Obviously, recycled Ni component is well-distributed on the catalyst surface. In fact, Ni° plays a role in Ni/Al catalyst for NO$_x$ conversion. The more Ni sites exists on the surface, the earlier NO$_x$ conversion starts. It was evidenced that the catalyst 20Ni/Al and 30Ni/Al continuously show the earlier NO$_x$ conversion beginning in accordance with 5Ni/Al and 10Ni/Al samples. However in cases of 20Ni/Al and 30Ni/Al, increasing too much active sites on the support can cause obscuring and agglomeration phenomena, thereby reducing available active sites of the catalyst. The Ni contribution induce decreases in the specific surface area and pore volume. It appears that samples with x = 5 and 10 decreases 8 and 14 m$^2$g$^{-1}$ in specific surface area and 0,01 and 0,04 cm$^3$g$^{-1}$ in pore volume, compared to non-Ni/Al$_2$O$_3$ sample. And they are up to 23 and 39 m$^2$g$^{-1}$ specific surface along with 0,05 and 0,09 cm$^3$g$^{-1}$ pore volume respective decreasing happened to samples with x = 20 and 30, compared to non-Ni/Al$_2$O$_3$ sample.

Among the catalysts prepared and tested, 10Ni/Al which content 10% Ni by weight has the only 116 m$^2$g$^{-1}$ surface area and 0,21 cm$^3$g$^{-1}$ pore volume (Table 1), but it has shown the maximum catalytic effectiveness of Ni sites in NO$_x$ dissociation and removal (reaches $T_{60}$ at 332°C, reaches 63% at 350°C and shows the highest efficiency with 98,5% at 471°C).
3.2. Effect of supporting sites to catalytic activity

3.2.1. Effect of Ba content to catalytic activity

The Ba component was aimed at increasing the defect as well as the adsorption affinity for NO\textsubscript{x} of catalysts. The Ba content is prepared on 10Ni/Al are 5\%, 10\%, 15\% and 20\% by BaO weight and denoted as 10Ni\text{y}Ba/Al (y = 5, 10, 15, 20 respectively). Results from the experiments shows that the catalyst with 10 wt\% Ba content exhibits the best NO\textsubscript{x} conversion. Compare to the non-Ba catalyst, the NO\textsubscript{x} performance finished promptly with the optimal efficiency is 98\% at 407°C (64°C sooner than 10Ni/Al); although the Ba addition neither start the conversion and reach T\textsubscript{60} value earlier nor achieve a higher performance at 350°C (63\%, equal to 10Ni/Al). In fact, the NO\textsubscript{x} performance is only enhanced after 350°C (the conversion rate is 0.61\%/°C compared to 0.29\%/°C in similar temperature period of 10Ni/Al). The BaO addition reduces the surface area massively (about 25\% of 10Ni10Ba/Al compared to 10Ni/Al); this can be the reason for non-improved catalytic activity in early stages of Ba modified samples. However, the XRD results show that 10Ni10Ba/Al has clearer Al\textsubscript{2}O\textsubscript{3} and Ni\textsuperscript{o} peaks than non-Ba samples. Hence, despite there is the surface area and pore volume smaller than non-Ba modified samples (decreased 29.1 m\textsuperscript{2}g\textsuperscript{-1} and 0.04 cm\textsuperscript{3}g\textsuperscript{-1} respectively) , the 10Ni10Ba/Al sample has a better distribution of Ba and Ni on surface.

Noticeably, this result was approximate to the drawing of Choi et al in which their 2Pt20Ba/Al\textsubscript{2}O\textsubscript{3} catalyst shows 65\% NO\textsubscript{x} conversion at the same temperature (350°C) [8]. Therefore, it is no doubt to say that with merely two catalytic components Ni and Ba coated, the modified Ni catalyst shows their competition to traditional precious ones.

3.2.2. Effect of Mn content to catalytic activity

Mn is added to the catalyst 10Ni10Ba/Al with the content of 5\%, 10\%, 15\% and 20\% by MnO\textsubscript{2} weight and denoted as 10Ni10BazMn/Al (with z = 5, 10, 15, 20 respectively). With the amount of 10 wt\% Mn, the catalyst is improved marvelously compared to non-Mn modified catalysts (lower beginning temperature, T\textsubscript{60} reached sooner, the maximum efficiency is 98\% at only 350°C). The XRD results (Figure 3) show that the MnO\textsubscript{2} peaks of 10Ni10Ba10Mn/Al sample have the highest intensity that can express a large amount of Mn supporting sites on the catalyst surface.
Figure 3. X-ray diffraction pattern of the 10Ni10Ba5Mn/Al catalyst - (°) Ni, (+) γ-Al2O3, (*) BaAl2O4, (Δ) MnO2

The Mn component expectedly increases anion oxide defected sites and improves heat resistance for catalyst [14]. As expected, the modification with Mn enhances the catalytic activity significantly in most of values. By comparison with non-Mn modified samples, the catalytic performance starts earlier (83°C earlier), reaches $T_{60}$ at a lower temperature (36°C lower), and better still complete the conversion at only 350°C (98%, 47°C sooner). This results are well suited to reports by Iwamoto [14], which will help dissociated adsorption of NOx takes place better on active Ni sites as well as supporting Mn sites. Compare to a related study of Nguyen et al with the Pt/20BaMn/Al catalyst at an approximate temperature, the NOx conversion performance of this study is considerably elevated (98% vs 74-84%) [5]. Although the Mn addition makes a considerable decrease in the surface area (17,1 m²g⁻¹), it only has a slight decline in the pore volume (0,02 cm³g⁻¹, Table 1), so as the structure of Mn modified catalysts are more favorable than the previous.

Table 1. Physical properties and characteristics of the NOx conversion process of tested catalysts

|       | Begin at (°C) | $T_{60}$ (°C) | At 350°C (%) | Max. (%) at (°C) | $S_{B.E.T.}$ (m²g⁻¹) | Pore vol. (cm³g⁻¹) |
|-------|---------------|---------------|--------------|------------------|----------------------|-------------------|
| Al    | /             | /             | /            | /                | 130                  | 0,25              |
| 5Ni/Al| 260           | 494           | 16           | 68,500           | 122                  | 0,24              |
| 10Ni/Al| 203          | 332           | 63           | 98,471           | 116                  | 0,21              |
| 20Ni/Al| 183          | 354           | 59           | 95,500           | 107                  | 0,20              |
| 30Ni/Al| 188          | 398           | 43           | 94,500           | 91                   | 0,16              |
| 10Ni5Ba/Al| 220      | 362           | 54           | 96,428           | 109                  | 0,21              |
| 10Ni10Ba/Al| 198     | 343           | 63           | 98,407           | 86,9                 | 0,17              |
| 10Ni15Ba/Al| 196     | 362           | 53           | 90,445           | 75,4                 | 0,14              |
| 10Ni20Ba/Al| 211     | 379           | 43           | 95,485           | 59,2                 | 0,12              |
| 10Ni10Ba5Mn/Al| 209   | 383           | 44           | 62,385           | 73,5                 | 0,15              |
| 10Ni10Ba10Mn/Al| 115    | 307           | 98           | 98,350           | 69,8                 | 0,15              |
| 10Ni10Ba15Mn/Al| 193    | 381           | 41           | 60,381           | 65,7                 | 0,13              |
| 10Ni10Ba20Mn/Al| 209    | /             | 41           | 55,376           | /                    | /                 |
| 10Ni10Ba5Ce/Al| 146   | 321           | 72           | 85,387           | 77,4                 | 0,14              |
| 10Ni10Ba10Ce/Al| 158    | 295           | 79           | 99,416           | 77,0                 | 0,13              |
| 10Ni10Ba15Ce/Al| 162    | 335           | 64           | 84,365           | 73,5                 | 0,13              |
| 10Ni10Ba20Ce/Al| 179    | 396           | 49           | 80,411           | /                    | /                 |
3.2.3. Effect of Ce content to the catalytic activity

Beside Mn component which creates a favorable defection on catalyst surface, another component is popularly used in raising NO\textsubscript{x} conversion is Ce. According to involved reports, the CeO\textsubscript{2} addition could help other active and supporting sites to be dispersed better on support, creates oxygen trap sites so as increases the product selectivity for N\textsubscript{2} by shifting the balance of NO $\leftrightarrow$ O\textsubscript{2} + N\textsubscript{2} reaction to the forward direction [6, 15, 23]. To compare separately the effectiveness of Ce and Mn in changing the physical structure of the catalyst, CeO\textsubscript{2} is added to the sample 10Ni10Ba/Al. With the content of 5%, 10%, 15%, 20% by CeO\textsubscript{2} weight, the samples are denoted as 10Ni10BatCe/Al (with $t = 5, 10, 15, 20$ respectively).

The modification with Ce alters catalytic activity and the best improvement is performed by sample 10Ni10Ba10Ce/Al with (Table 1) with 79% NO\textsubscript{x} concentration is converted. These results was also more favourable than the drawing of Ha et al that their 15Ni10CeBa/Al catalyst shows only 38% NO\textsubscript{x} conversion at the same temperature (350°C) [4].

By comparison with Ba-modified catalyst, the NO\textsubscript{x} conversion starts earlier (40°C), reaches T\textsubscript{60} at a lower temperature (48°C), and gives a higher 350°C conversion efficiency (16%). However, the conversion completes later than the non-Ce sample (9°C, at 99%). The physical structure analysis results (Table 1) show that the Ce addition reduces both surface area and pore volume of the catalyst (respectively 9.9 m\textsuperscript{2}g\textsuperscript{-1} less and 0.04 cm\textsuperscript{3}g\textsuperscript{-1} slightly more than the Mn modified samples). The catalytic improvement (through the better metals dispersion on support) of Ce modified samples taken at the same range of temperature is also agreed by Zhu et al [15].

Nevertheless, the improvement is not more impressive when brought into comparison with 10Ni10Ba10Mn/Al. Although the 10Ni10Ba10Ce/Al reached T\textsubscript{60} value earlier (12°C earlier), the efficiency at 350°C is only 79% which is 98% by the Mn modified catalyst, worse still the conversion complete much later than both Ba-Mn modified and Ba modified samples.

![Figure 4](image_url)

**Figure 4.** NO\textsubscript{x} conversion vs temperature light-offs over modified catalysts

3.3. The correlation of NO\textsubscript{x} conversion capabilities of recovered Ni and trade Ni catalysts

Some catalysts are prepared from commercial Ni(NO\textsubscript{3})\textsubscript{2} instead of recovered Ni solution with the similar component ratio as the 10Ni10Ba10Mn/Al sample (denoted as 10Ni\textsubscript{nc}10Ba10Mn/Al). The NO\textsubscript{x} light-off comparative results of these catalysts is shown in **Figure 5**. It can be seen that there is a slightly difference between these catalyst versions’ performance. By comparison with the recovered Ni catalyst (○), the commercial Ni catalyst (□) takes a conversion which starts earlier (7°C), reaches T\textsubscript{60} at a lower temperature (2°C), gets a higher efficiency at 350°C (2%). Due to these extent
equivalence, it is capable of putting recovered Ni from fertilizer plants to good use for NO\(_x\) conversion.

![Figure 5. NO\(_x\) conversion vs temperature light-offs over 10Ni10Ba10Mn/Al and 10Ni\(_{10}\)Ba10Mn/Al catalysts](image)

4. Conclusion
Among the prepared and tested catalysts, the catalyst 10Ni10Ba10Mn/Al shows the earliest conversion beginning (115°C), reaches \(T_{60}\) value promptly (307°C) and achieves the highest performance of 98% at 350°C. This sample has the surface area of 69.8 m\(^2\)g\(^{-1}\) and 0.15 cm\(^3\)g\(^{-1}\).

In the performance of the catalyst 10Ni10Ba10Mn/Al, the NO\(_x\) conversion mechanism is very complex, including the reduction, trap and direct thermal-decomposition. The main components of catalyst include active Ni sites which promote the NO\(_x\) direct thermal-decomposition process at low temperature due to enhancing the dissociative adsorption of NO molecules on the surface of the catalyst; Ba sites are capable in trapping NO\(_x\) molecules, support for the NO\(_x\) reduction by CO; Mn sites also support for the NO\(_x\) absorption on the catalyst surface through oxygen defects and are potential to improve heat resistance for catalyst.

The use of recovered Ni derived from waste catalyst of fertilizer plants instead of commercial Ni to prepare catalyst for NO\(_x\) treatment is highly promising. The comparative results in NO\(_x\) conversion of those kind of Ni catalysts show a very little different (93% and 95% at 350°C, respectively).

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