

出國報告（出國類別：國際研討會）

## 出席 2012 年亞太環境科學與技術會議 心得報告

服務機關：國防大學理工學院化學及材料工程學系

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## 摘要

2012 年亞太環境科學與技術會議，由 APEST2012 組織委員會(University Of Malaya and Nanchang University)信息工程研究學會籌辦，會議地點為馬來西亞吉隆坡，於 2012 年 2 月 1 - 2 在吉隆坡 Impiana Hotel (迎碧安娜酒店)舉行。本次會議之主題為全球環境變化和生態系統管理、遙感應用及空間信息系統的應用，台灣有 5 位師、生代表於會議中提出論文報告，內容涵蓋環境修護和生態工程、環境力學、圖形和圖像處理、科學計算可視化、GIS 方法和應用、遙感圖像處理的方法等，提供參與學者們一個溝通互相觀摩學習的機會，雖為小規模之會議，會議籌備尚稱周詳，使大會顯得熱鬧非凡，賓主盡歡。其中台灣地區則有包括國防大學、屏東科技大學、陸軍專科學校等，共計 5 員。在會議中聆聽學者之學術演講，其中 Qing Xin 利用脈衝光進行苯酚之降解研究，並依實驗數據推導苯酚降解之反應機制，領域與我們較相近，對我們的啟發及受益最大。本次會議提供一個相當好的學術交流平台，藉由此交流進而瞭解國際未來研究發展方向及趨勢，真是獲益匪淺。此外，藉由與大會各專家學者之交換研究心得及吸取他人寶貴之研究經驗，將可做為本實驗室日後研究之參考。

筆者感謝獲國科會經費補助成行，發表題目為“Rapid Degradation of Phenol under Microwave-assisted over Nickel Oxide”，達到與專家學者切磋交流的機會，真是獲益匪淺。

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## 壹、會議目的

2012年亞太環境科學與技術會議，由 APEST2012 組織委員會(University Of Malaya and Nanchang University)信息工程研究學會籌辦，為亞太地區在環境科學技術領域之重要學術會議，其會議宗旨在於結合亞太地區國家有關全球環境變化和生態系統管理、遙感應用及空間信息系統的應用研究領域之學者專家，就專長領域進行系列學術研究成果發表及新知討論，歷年來相關之研討會對學術領域有深遠的影響及貢獻。因此，藉由此學術交流進而瞭解國際未來研究發展方向及趨勢，並與各國專家學者交換研究心得及吸取他人寶貴之研究經驗，將可做為日後研究之參考。

## 貳、會議過程

(一)本會議屬於小型的研討會，參加人員計有台灣5位師、生代表及亞太地區國家約60位代表於會議中提出論文報告，除論文口頭報告，亦包含壁報論文。會議於2012年2月1 - 2在馬來西亞吉隆坡Impiana Hotel (迎碧安娜酒店)舉行。大會所發表的論文範圍很廣，包含環境修護和生態工程、環境力學、圖形和圖像處理、科學計算可視化、GIS方法和應用、遙感圖像處理的方法等，與會者多為該領域學有專精之教授與學者，於研討會期間與各學者相互密切交流之下獲益良多。

(二)會議行程之安排為一天口頭報告(09:00 - 18:00)、一天自由發言討論及壁報論文(會議行程詳如下表)。為能使發表論文的學者有機會與其他學者們深入討論，每場之研討時間均欲罷不能而超出排定之時段，以便能詳細解說討論與互換研究心得，效果非常好。另大會別於第二天安排自由發言時段，期能針對第一天所激盪之主題或需進一步深入了解之內容，再一次讓有興趣的專家學者能面對面研討、爭辯、溝通。在聆聽及交流過程中可體會各與會學者對研究之投入是非常的專注，並了解所有的研究成果均是心血結晶的累積。

2012 Asia Pacific Conference on Environmental Science and Technology (APEST 2012)

Date/Time	February 1 (Wednesday)	February 2 (Thursday)
09:00 - 09:15	Open Ceremony	Free Talk Poster Presentation
09:15 - 10:15	Keynote Speech (I) Prof. Ben Kwang-Mong SIM	
10:00 - 10:15	Coffee Break	
10:15 - 11:00	Keynote Speech (II) Prof. Gerald Schaefer	
11:00 - 12:00	Oral Session (I)	
13:00 - 15:20	Oral Session (II)	
15:20 - 15:40	Coffee Break	
15:40 - 18:00	Oral Session (III)	

## 參、會議心得

本研討會規模雖小，卻也結合了六個領域的內容：環境修護和生態工程、環境力學、圖形和圖像處理、科學計算可視化、GIS方法和應用、遙感圖像處理的方法等，會議是由University of Malaya and Nanchang University的教授籌辦。參加人員為亞太地區之專家學者，共計發表論文約150篇，並刊載於Advances in Biomedical Engineering期刊。大會所討論之範圍很廣，部份足以為我國所參考之依據，另相關論述主題亦十分具參考價值。

於研討會中，透過各學者不同領域的經驗，於問答間各取所需，達到智識精進功效，並積極與各學者交換演講意見達到學術交流目的。經過此次研討會歷練，使本人對未來之研究更具信心，將持續於此領域探討研析，並且對於後續之研究將會秉持精益求精的精神戮力完成。本次會議提供一個相當好的知識交流平台，藉由此學術交流進而瞭解未來研究發展方向及趨勢，真是獲益匪淺。

## 肆、建議事項

筆者有感於研究技術日新月異，應多與各國之研究學者專家交流，才能以獲裨益。願以此次的與會心得與大家分享共勉之，本次研討會攜回會議論文集資料。

作者發表之論文

Rapid Degradation of Phenol under Microwave-assisted over Nickel Oxide

## Rapid Degradation of Phenol under Microwave-assisted over Nickel Oxide

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### Abstract

The microwave-enhanced catalytic decomposition (MECD) of phenol using nickel oxide was studied. A nickel hydroxide, Ni(OH)<sub>2</sub>, was prepared by the microwave-assisted heating technique from a nickel nitrate aqueous solution and sodium hydroxide. Then, the nickel hydroxide was oxidized by liquid oxidation with sodium hypochlorite. The nickel oxide was characterized by X-ray diffraction (XRD), infrared spectroscopy (IR), temperature-programmed reduction (TPR) and scanning electron microscopy (SEM). The catalytic activities towards the decomposition of phenol on the efficiency of the degradation were further investigated under the continuous bubbling of air through the liquid phase and quantitatively evaluated by high pressure liquid chromatography (HPLC). The catalyst morphology and its composition were found to be changed after the catalytic reaction. The decomposition rate was increased by 5.638 times in the presence of air. Increasing the catalyst loading from 0.03 g to 0.15 g increased the removal efficiency of phenol from 32% to 95%.

**Keywords:** Nickel oxide; Microwave; Degradation of phenol; Catalytic decomposition.

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## 1. Introduction

The organic pollutants in industrial wastewater are of serious concern. Hazardous organic pollutants, such as aromatic pollutants, in particular phenol, chlorophenol compounds and phenolic derivatives, have been on the EPA's priority pollutants list since 1976 [1]. These compounds are considered hazardous because of their potential to harm human health. Indeed, it is necessary to eliminate them from industrial wastewater before it is discharged. One of the most promising techniques to eliminate such pollutants is complete oxidative mineralization by heterogeneous photocatalysis [2-4]. Sonochemical degradation [5, 6], an advanced oxidation process with UV/H<sub>2</sub>O<sub>2</sub> (AOP) [7], catalytic oxidation [8] and microwave-enhanced oxidation processes [9, 10] have received considerable attention in recent years as regards the removal of these hazardous pollutants. One of the most promising technologies is utilizing the microwave-enhanced photocatalysis method for the degradation of 4-chlorophenol [11]. The use of microwaves as a source of energy has grown as it has become more economical.

Nano-sized particles are considered particularly attractive for catalysis due to their high reactivity which is attributed to the enhanced surface area [12]. An environmentally friendly method for destroying organic pollutants in aqueous solutions is based on heterogeneous catalytic reactions providing complete oxidation. Through the use of the appropriate catalytic systems and reaction conditions, a high selectivity towards environmentally harmless products can be achieved at mild conditions (room temperature and atmospheric pressure) [13]. The most common catalysts, such as nickel oxide and cobalt oxide have also been shown to degrade contaminants [14, 15].

In a previous work, we successfully achieved the decomposition of phenol with the microwave-enhanced catalytic degradation (MECD) method [16]. For this reason, the present work has focused on the MECD method for the degradation of phenol. In this study, we examined the activity of nickel oxide nanoparticles as catalysts for the

degradation of organic pollutants in water. Therefore, the major objective of this study was adopting nickel oxide as the active component and using microwave irradiation technology to promote the decomposition of phenol.

## 2. Experimental

50 ml of 0.6 M  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  solution was added to the 100 ml of 3.2 M NaOH solution. The reaction was carried out for 10 min in air in a microwave apparatus (100 W, 2450 MHz, CEM, USA). The obtained as-prepared nickel hydroxide,  $\text{Ni}(\text{OH})_2$ , presented as a green colloid solution. Then, the as-prepared nickel hydroxide was oxidized by liquid oxidation with sodium hypochlorite drop by drop under constant stirring at  $0^\circ\text{C}$  for 2 h. Finally, a kind of black sediment was formed. The as-prepared sample was thoroughly washed with distilled water six times and dried at  $100^\circ\text{C}$  for 16 h.

X-ray diffraction (XRD) measurements were performed using a MAC Science MXP18 diffractometer with  $\text{Cu K}_{\alpha 1}$  radiation ( $\lambda = 1.5405 \text{ \AA}$ ) at 40 kV and 30 mA with a scanning speed in  $2\theta$  of  $4^\circ \cdot \text{min}^{-1}$ . The reduction behavior of the nickel oxide was studied by temperature-programmed reduction (TPR). About 50 mg of the sample was heated in a flow of 10%  $\text{H}_2/\text{N}_2$  gas mixture at a flow rate of  $10 \text{ ml} \cdot \text{min}^{-1}$ . During TPR, the temperature was increased by  $7^\circ\text{C} \cdot \text{min}^{-1}$  increment.

The MECD experiments for the decomposition of the phenol were carried out in a thermostated static microwave apparatus (CEM. Discover, USA, 2450 MHz, 300 W) upon continuous stirring. 60 ml of aqueous phenol solution was used for each experimental run. Air was bubbled into the solution for 30 min before adding the catalyst. Then, a fit amount of catalyst was suspended in the solution. The air was continuously bubbled during the runs. To draw 3-5 ml the upper layers of suspension after static 10 min and filtrated with syringe filters for each experimental data for quantitative analysis with HPLC. An absorbance of 270 nm was used to measure the concentration of phenol.

### 3. Results and Discussion

#### 3.1 Decomposition of phenol

The effect of dissolved oxygen on the decomposition of phenol ( $200 \text{ mg}\cdot\text{L}^{-1}$ ) by the MECD method over the nickel oxide catalyst was investigated under  $\text{pH} = 7$  and  $T = 40^\circ\text{C}$ . The results [Fig. 1] showed that the dissolved oxygen clearly enhanced catalytic efficiency in the presence of nickel oxide, increasing the first-order decomposition rate by 5.638 times. It seemed reasonable to suggest that the reaction only proceeded on the catalyst surface, and not homogeneously. Additional evidence for this conclusion was the fact that, after removing the catalyst from the reaction mixture, the degradation process almost ceased.

The catalyzed decomposition of phenol using various loadings of nickel oxide was then studied. As shown in Fig. 2, increasing the nickel oxide dosage increased the decomposition of phenol when the concentration of dissolved oxygen was constant, indicating that the dominant oxidant in the reaction increased when the catalyst dosage was increased. Increasing the catalyst loading [Fig. 2(a)] from 0.03 to 0.06, 0.09, 0.12 and 0.15 increased the phenol removal efficiency from 37% to 86%, 95%, 98% and 100%, respectively, at a reaction time of 30 min. The rate constant [Fig. 2(b)] went from  $0.013 \text{ min}^{-1}$  to  $0.064 \text{ min}^{-1}$ ,  $0.097 \text{ min}^{-1}$ ,  $0.112 \text{ min}^{-1}$  and  $0.210 \text{ min}^{-1}$ , respectively. At the range of loading, the removal of phenol increased linearly with the catalyst loading; however, the presence of excess catalyst in the aqueous solutions did not cause a shielding effect on the penetration light, suggesting that the factors affecting the microwave-enhanced catalytic decomposition were not only microwave but also thermally induced.

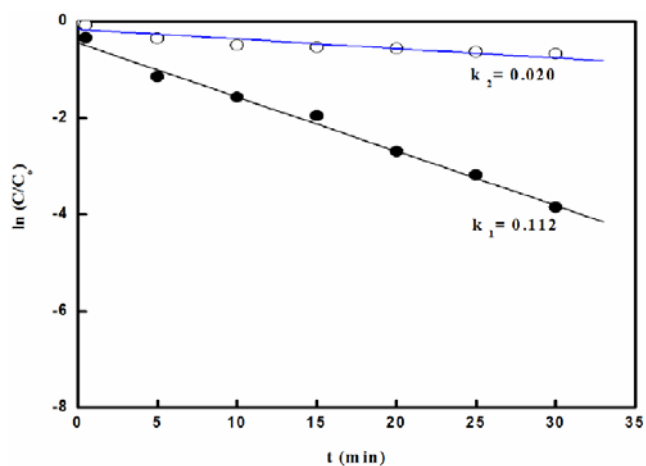


Fig. 1 Comparison of the degradation of phenol by MECD method over nickel oxides (0.12 g) under pH = 7,  $C = 200 \text{ mg}\cdot\text{L}^{-1}$  and  $T = 40 \text{ }^\circ\text{C}$ : (●) with air; (○) without air.

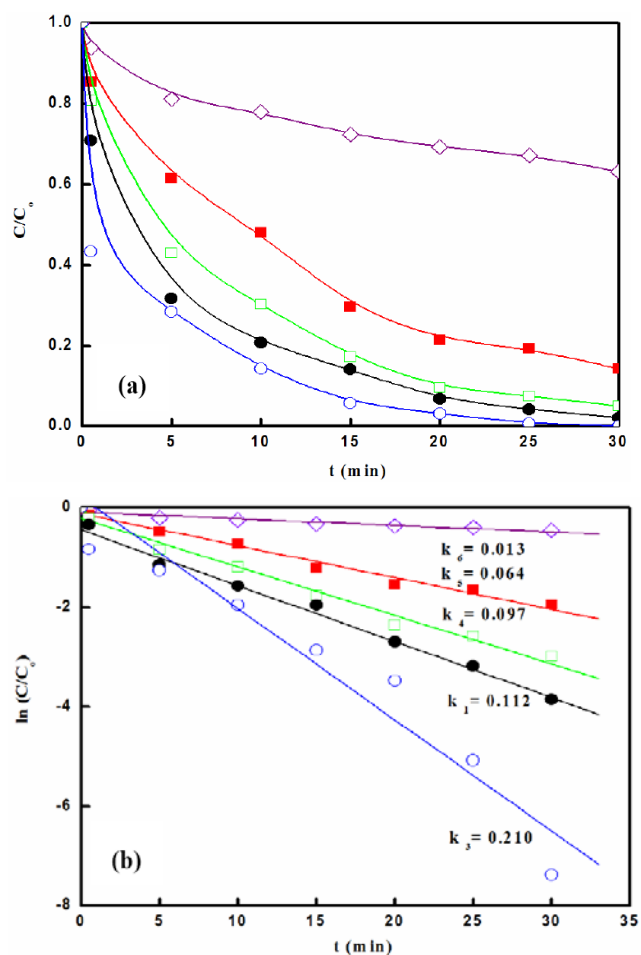


Fig. 2 (a) The effect of catalyst dosage on the decomposition of phenol by MECD method (b) plot of  $\ln(C/C_0)$  vs. time at  $T = 40 \text{ }^\circ\text{C}$  and  $C = 200 \text{ mg}\cdot\text{L}^{-1}$ : (○) 0.15 g; (●) 0.12 g; (□) 0.09 g; (■) 0.06 g; (◇) 0.03 g.

The experiments with reused catalyst and without catalyst were carried out under identical reaction conditions [Fig. 3]. In the microwave-enhanced catalytic decomposition experiments, rate constants of  $0.112 \text{ min}^{-1}$ ,  $0.026 \text{ min}^{-1}$  and  $0.003 \text{ min}^{-1}$  for the fresh, used and without catalyst experiments were observed at a reaction time of 30 min. Experiments both with and without nickel oxide were studied in order to determine its role in the decomposition of phenol: with nickel oxide, the decomposition rate increased by 37 times; the use of a catalyst caused the decomposition rate to decrease 4 times.

The pH values of different wastewaters will influence the microwave-enhanced catalytic decomposition reactions for the removal of the pollutants. Similarly, the pH value plays an important role in the degradation of phenol. In this study, the effect of pH was examined by adjusting the initial pH of the reaction mixture in the range of 4-10. From Table 1 it can be seen that the microwave-enhanced catalytic decomposition efficiency of phenol increased in acidic or alkaline mediums. The decomposition efficiency (rate constant) of phenol was 80% ( $0.040 \text{ min}^{-1}$ ) at pH 10, 97% ( $0.040 \text{ min}^{-1}$ ) at pH 4 and 98% ( $0.040 \text{ min}^{-1}$ ) at pH 7. Christoskova et al. [15] showed that at  $\text{pH} \geq 10$  no oxidation of phenol occurred even with a high concentration of the catalyst or temperature ( $115^\circ\text{C}$ ) over the nickel oxide. We, however, were now in the position to suggest that microwave irradiation efficiently enhanced the catalysis oxidation of phenol over nickel oxide.

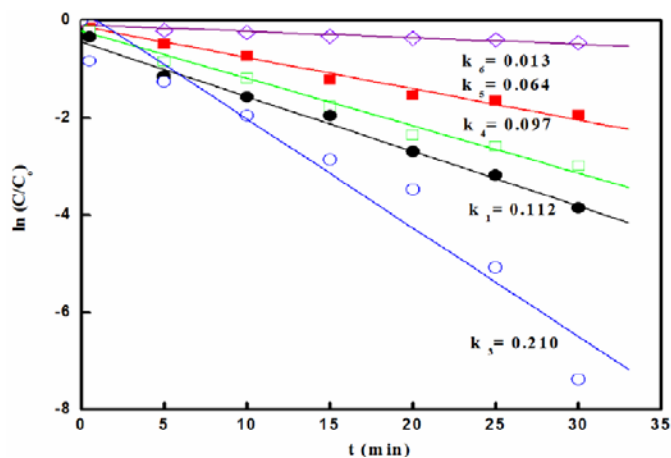


Fig. 3 The effect of catalyst recycle on the decomposition of phenol by MECD method at  $T = 40^\circ\text{C}$  and  $C = 200 \text{ mg}\cdot\text{L}^{-1}$ : (●) fresh; (○) used; (■) without catalyst.

Table 1 Influence of pH, temperature and initial concentration on the degradation of phenol

pH	Temperature (°C)	Initial concentration (200 mg · L <sup>-1</sup> )	Degradation of phenol	
			$\alpha_{30}^*$ (%)	$k$ (min <sup>-1</sup> )
4	40	200	97	0.092
7	40	200	98	0.119
10	40	200	80	0.040
7	55	200	97	0.093
7	70	200	90	0.064
7	40	100	100	0.141
7	40	50	100	0.449
7	40	25	100	0.495

\*Degree of phenol conversion is determined at 30 minute.

To determine the effect of temperature on reaction rates, several runs were performed at temperatures between 40 and 70°C. Table 1 shows the results of three runs conducted at pH 7 with an initial phenol concentration of 200 mg·L<sup>-1</sup>. We found that heating did not enhance the oxidation rate due to the properties of microwave absorption. The ability of a substance to convert electromagnetic energy into heat at a given frequency and temperature can be determined by the dielectric loss tangent ( $\tan \delta$ ). Temperature has a great influence on the measurement of  $\tan \delta$  [17, 18]. The scheme of phenol oxidation has been carried out under analogous conditions as reported by Lai et al. [16]. Microwave absorbance under low temperature generated more microwaves and enhancing catalysis which led to the degradation of the phenol.

The microwave-enhanced catalytic decomposition of phenol at different initial concentrations in the range 25-200 mg·L<sup>-1</sup> was investigated. The rate constant decreased from 0.112 min<sup>-1</sup> to 0.495 min<sup>-1</sup>. The phenol at an initial concentration under 100 mg·L<sup>-1</sup> had completely decomposed at 30 min.

### 3.2 Characterization of nanosized nickel oxides

The crystal structure of nickel oxide (fresh and used) was examined by XRD, as shown in Fig. 4. As shown by the peak intensity of the diffractograms, this black powder had XRD patterns which corresponded to the peaks at ca. 19.0° and 38.4°, and these locations were similar to  $\beta$ -NiOOH with the peaks at the (001) and (002) planes. In comparing the XRD of fresh catalyst with used catalyst, double phases of  $\beta$ -NiOOH and  $\beta$ -Ni(OH)<sub>2</sub> (JCPDS 14-0117) were obtained. In the study of the oxidation of cyclohexanol, Yi et al. [19] found that the used NiOOH catalyst transferred to Ni(OH)<sub>2</sub>. We confirmed that part of the catalyst on the degradation of phenol reduced to  $\beta$ -Ni(OH)<sub>2</sub>.

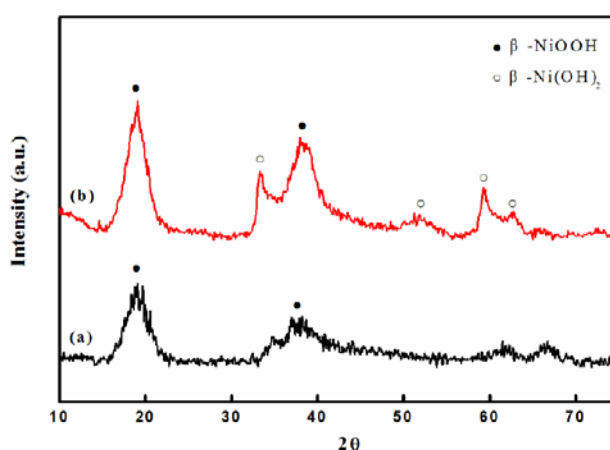


Fig. 4 XRD pattern of nickel oxide catalyst: (a) fresh (b) used.

In order to understand whether the content of Ni<sup>3+</sup> over the fabricated nickel oxide participated in and promoted the activity necessary for the decomposition of phenol, the analysis of TPR for the used catalyst (after 30 min in the decomposition of phenol at 40°C) proceeded. Reduction profiles of the fresh nickel oxide [Fig. 5(a)] showed two reduction peaks similar to those observed in the supported nickel catalysts [20, 21]. These profiles pointed to a two-step reduction process: the first step was low intensity at 160°C, and the more intense second step whose maximum occurred at 280°C. In a comparison of the TPR of the fresh [Fig. 5(a)] with the used [Fig. 5(b)] nickel oxide catalyst, the shift of  $T_{\text{red}}$  for  $R_1$  and  $R_2$  accompanied by the decreasing of the  $R_1$  area further suggested that the surface active oxygen initiated the catalytic oxidation of phenol at about 40°C.

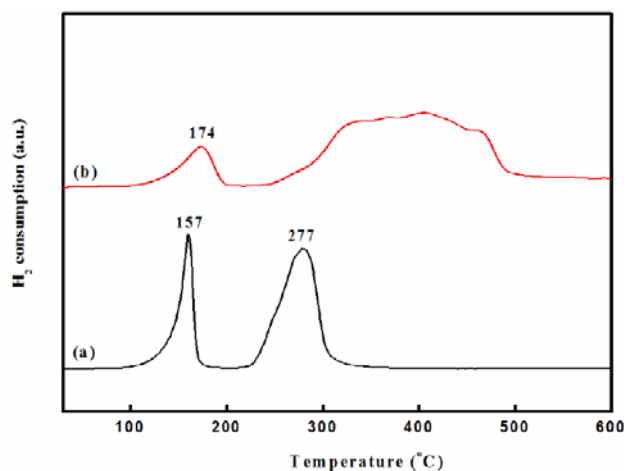


Fig. 5 TPR profile of nickel oxide catalyst: (a) fresh (b) used.

#### 4. Conclusions

An environmentally friendly process on the degradation of phenol was successfully developed.

- (1) Nickel oxide was found to be a useful catalyst for the decomposition of phenol in water.
- (2) Phenol was degraded 98% by the MECD method within 30 min under pH = 7, T = 40°C and C = 200 mg·L<sup>-1</sup> over the nickel oxide catalyst.
- (3) The activity of the phenol thus degraded was strongly dependent on the oxidation state of nickel.

#### 5. Acknowledgement

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