



# Adsorption and bio-sorption of nickel ions and reuse for 2-chlorophenol catalytic ozonation oxidation degradation from water



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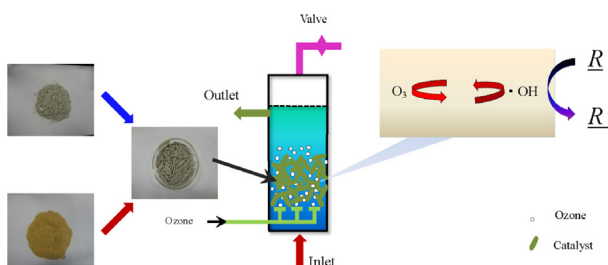
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## HIGHLIGHTS

- Biomass and fly ash which were widespread for adsorption of heavy metal ions.
- Preparation of catalyst by saturated adsorbents for 2-chlorophenol ozone degradation.
- This work demonstrated that the O<sub>3</sub>/catalyst process was an effective pathway.
- The use of nickel ions, fly ash and sawdust to achieve the recycling utilization of resources.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

### Article history:

Received 31 August 2013

Received in revised form 4 November 2013

Accepted 6 December 2013

Available online 12 December 2013

### Keywords:

Adsorption  
Fly ash  
Sawdust  
Catalyst  
2-Chlorophenol  
Catalytic ozonation

## ABSTRACT

This work explored the preparation of an effective and low-cost catalyst and investigated its catalytic capacity for 2-chlorophenol ozonation oxidation degradation in wastewater by using an ozone oxidation batch reactor. The catalyst was directly prepared by the reuse of fly ash and sawdust after saturated adsorption of nickel ions from wastewater, which was proposed as an efficient and economic approach. The obtained catalyst was characterized by TGA, BET, FTIR, XRD, and SEM, the results showed that fly ash as the basic framework has high specific surface area and the addition of sawdust as the porogen agent could improve the pore structure of the catalyst. The adsorption of nickel ions by fly ash and sawdust from aqueous solution was also investigated in this study. The results obtained from the experiments indicated that adsorption of nickel ions by fly ash and biomass sawdust could be well described by Langmuir isotherm model and pseudo second order kinetic model. The catalytic performance of catalyst was studied in terms of the effect of time, liquid–solid ratio and pH on 2-chlorophenol ozonation degradation. It was found that the catalyst could effectively improve the ozonation reaction rate at pH = 7 with a 2:1 liquid–solid ratio. The kinetic study demonstrated that the reaction followed the first order model, and the rate constant increased 267% (0.03–0.1 min<sup>-1</sup>) of 2-chlorophenol ozonation degradation with 5 mmol/L concentration at pH = 7.0 compared with ozonation alone.

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## 1. Introduction

The pollution from heavy metals has attracted widespread attention due to their toxicity, persistency and non-degradability in the ecosystem [1,2]. Several technologies have been adopted

for the removal of heavy metals from aqueous solutions, such as ion exchange, chemical precipitation, membrane filtration, electrodeposition, reverse osmosis, and adsorption [3]. The adsorption or bio-sorption have received much attention in recent years due to its properties which used in wastewater treatments with the advantages of simplicity in operation, low cost, and insensitivity to toxic substances compared to other separation methods. Thus, many researches focus on seeking cost-effective adsorbents such as rice husk, grapefruit peel, peat, activated carbon, zeolite, and fly ash

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to deal with these issues. In addition, abundant of bio-sorbents such as agricultural wastes, microorganisms, algae and plants have been reported for the effective removal of metal ions from aqueous solutions. It has many possible applications in wastewater treatment field and also been recognized as potential engineering material, which offers economic benefits [4–6].

Furthermore, many researches focus on the adsorption of heavy metal nickel ions while a little attention has been paid to the further utilization of the saturated adsorbent. The proposed approach to reuse the adsorbents or bio-sorbents is washed by acid solutions. Zheng et al. [7] reported a desorption process in which the adsorbent were immersed into 0.1 M HCl solution for several times then washed by 0.2 M NaOH and deionized water, finally dried for recycle. In this process, some activated groups may release and result in the decrease of adsorption capacity.

On the other hand, the transition metal ions, such as nickel ions, have catalytic oxidation performance in organic wastewater treatment. Saeed et al. explored the preparation of nickel hydroxide catalyst and investigated its catalytic activities for oxidative degradation of phenol in aqueous medium using batch reactor [8]. However, little attention has been paid to the reuse of saturated adsorbent to prepare catalyst for organic waste water oxidation treatment.

As a strong chemical oxidant, ozone is widely used to convert nitrobenzene, oxalic acid, and other relatively bio-refractory organic contaminants into CO<sub>2</sub> and inorganic ions in water [9–12]. However, applications of ozonation in wastewater treatment are limited due to the major disadvantages of high cost, high selectivity of its oxidative power in ozonation (O<sub>3</sub> system). Consequently, various advanced oxidation processes (AOPs) have been developed as the potential methods for organic compounds degradation, such as O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>, UV/O<sub>3</sub>, UV/H<sub>2</sub>O<sub>2</sub>, Fenton and UV/Fenton reagents, photo-catalytic oxidation with TiO<sub>2</sub>, wet air oxidation, wet peroxide oxidation processes, electron beam and catalytic ozonation [13]. Catalytic ozonation is considered as a promising method for pollutant control because of its environmental friendliness and high efficacy. Catalytic ozonation can be homogeneous or heterogeneous, the latter being more applicable to wastewater treatment [8]. Wang et al. prepared a series of Ni catalysts supported on fly ash (FA) treated by various chemical methods and tested them in CO<sub>2</sub> reforming of methane for syngas production [14,15].

Therefore, the objective of this investigation are: (1) to explore the potential of fly ash and sawdust as low cost adsorbent for the treatment of nickel ions wastewater, (2) to prepare catalyst by adsorbed fly ash and sawdust and to characterize the catalyst by X-ray diffraction (XRD), Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR) and nitrogen adsorption-desorption experiment (BET), (3) to further evaluate the ozonation oxidation of 2-chlorophenol with the catalyst, (4) to study the degradation kinetic and mechanism of 2-chlorophenol ozonation oxidation. It is expected that the preparation of catalyst by fly ash and sawdust after adsorption would draw more attention to catalytic ozonation technology and push forward to its development in organic wastewater treatment.

## 2. Experimental

### 2.1. Chemicals and materials

2-chlorophenol, potassium iodide, potassium ferricyanide, nickel sulfate hexahydrate, 4-aminoantipyrine, supplied by Kermel, Tianjin, were used to prepare the concentration of 5 mmol/L of 2-chlorophenol, 20% potassium iodide solution, 8% potassium ferricyanide solution, 500 mg/L nickel sulfate solution, 2% 4-aminoantipyrine. Sodium hydroxide and hydrochloric acid

purchased from Dalian (China) were used to prepare 0.1 mol/L sodium hydroxide and hydrochloric acid solution. All the reagents were of analytical grade. All solutions were prepared with deionized water. The fly ash obtained from Inner Mongolia Autonomous Region (China).

Sawdust and fly ash were selected due to their good adsorptive properties and the presence of surface basic sites compared with chitosan, activate carbon, zeolite. The element analysis diagram of sawdust and fly ash was listed in Tables 1 and 2, respectively.

### 2.2. Adsorption of Ni(II)

The isotherm study of nickel ions adsorption was conducted by varying the initial concentration of nickel ions in aqueous solution ranging from 5 to 200 mg/L. 0.1 g adsorbent was stirred with 50 mL nickel ions solution at room temperature. Adsorption kinetics study was carried out with initial nickel ions concentration of 50 mg/L. A container with 2 g adsorbent and 1 L nickel ions solution was stirred at room temperature. And 1 mL of the solution was taken from the solution to measure the nickel ions concentrations in a certain time intervals.

The adsorption capacity (mg/g) was determined by the equation as follows:

$$q = \frac{(c_0 - c) \times V}{m} \quad (1)$$

where  $q$  was the nickel adsorption amount per mass fly ash and sawdust (mg/g),  $c_0$  was the initial concentration of nickel ions (mg/L),  $c$  was the concentration of nickel ions after adsorption (mg/L),  $m$  was the mass of fly ash and sawdust added (g), and  $V$  was the volume of solution (L).

### 2.3. Preparation of the catalyst

The fly ash and sawdust was dried at 120 °C for 12 h. A certain amount of fly ash and sawdust were added into 500 mg/L nickel sulfate solution. After nickel ions adsorption, the fly ash and sawdust was filtered, washed and dried at 100 °C, respectively. The fly ash and sawdust after nickel ions adsorption were evenly mixed with kaolin clay, sodium silicate and liquid paraffin, and then extruded by a banded extruder. The formed strip was dried at 120 °C for 10–12 h. Finally, the dried extruding catalyst was calcined at 500 °C for 4 h.

### 2.4. Characterization of the catalyst

TGA method was applied under program control temperature to measure the quality of the material along with the change of temperature (or time). Thermogravimetric measurements and differential thermal analysis were performed with a Perkin Elmer Diamond thermogravimetric differential thermal analyzer. The sample was placed in a platinum sample holder. Measurements in air with a flow rate of 100 mL min<sup>-1</sup> were carried out over a temperature range of 50–1000 °C with heating rate of 10 °C min<sup>-1</sup>.

Specific surface area of fly ash and the catalyst was determined by using a Quanta Chrome (Nova 2200e) surface area and pore size analyzer. Samples were degassed at 383 K for 20 min prior to analysis.

FTIR spectra were recorded in KBr medium using IR Prestige 21 (Bruker TENSOR 27 FTIR, Nicolet) in the range of 500–4000 cm<sup>-1</sup>.

X-ray diffraction (XRD) patterns were recorded using X-ray diffractometer (DX 2000 China), using CuK $\alpha$  radiation with a tube voltage of 40 kV and 20 mA with  $2\theta$  ranges from 10 °C to 70 °C.

SEM analyses were carried out by using Scanning Electron Microscope (JEOL-JSM-5600 LV Japan). For this purpose the

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