

# Sonocatalyst degradation of catechol from aqueous solution using magnesium oxide nanoparticles

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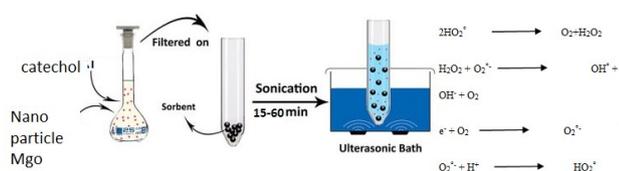
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## Graphical abstract



## Abstract

Catechol is a highly toxic phenolic organic compound used in various industries such as refineries, toxics, dyes and plastics. When this compound enters the environment, it can have negative effects on human health and the ecosystem. In this study-removed pyrocatechol from aqueous solutions using a sonocatalytic process using magnesium oxide nanoparticles. The study was conducted on a laboratory scale and the effects of pH, catalyst dose, initial concentration and reaction time under 130-kHz ultrasonic waves on the sonocatalytic degradation of pyrocatechol were investigated. The maximum process efficiency under optimal conditions (i.e. pyrocatechol concentration of 25 mg / l, pH = 3, nanoparticle dose of 500 mg / l after a contact time of 60 min) was 92%. The results showed that this process follows first order pseudokinetics. The results show that the sonocatalytic process in the presence of magnesium oxide nanoparticles has a high efficiency in removing catechol from aqueous solutions.

**Keywords:** Sonocatalytic, magnesium oxide, nanoparticles, catechol

## 1. Introduction

Available water resources are decreasing with the industrialization of the world, so new and innovative solutions for a safe water supply are on the rise. One of these solutions is the removal of organics from agricultural

wastewater, various industries such as refineries, toxins, dyes, and plastics. Catechol is a highly toxic phenolic organic compound used in various industries. Due to its high solubility in water, high toxicity and low degradability, as well as adverse effects on the environment and human health through red blood cell analysis, treatment of catechol wastewater is required. (Kermani *et al.*, 2016) According to the standards of the US Environmental Protection Agency (USEPA) and the World Health Organisation (WHO), the maximum permissible concentration of phenol and its derivatives in drinking water and industrial wastewater should not exceed 1 microgram per liter and 1 milligram per liter, respectively. (Bazrafshan *et al.*, 2019) recently, various methods for the removal of pollutants such as adsorption technology, zero reduction of reduced iron, electrochemical oxidation, advanced oxidation and wet catalytic oxidation of aqueous solutions have been investigated (Gogoi *et al.*, 2017). Compared to other available catalysts, MgO as an alkaline earth metal is considered as one of the most promising catalysts. Its notable features include low cost, ease of synthesis and non-toxicity to the environment (especially aquatic organisms). Additionally, the MgO nanocatalyst has high catalytic potential due to its large specific surface area; on the other hand, it has unique physicochemical reactivity and high thermodynamic stability.(Soltani *et al.*, 2017) Numerous studies have shown that ultrasound is a safe, effective and rapid method for destructing aromatics, phenols, chlorinated compounds and pesticides from aqueous solutions. (Wang *et al.*, 2015; Pili *et al.*, 2011) The general mechanism of the process in the oxidation of pollutants is the formation of tiny holes by sonic cavitation in the water. These holes are hot spots (temperature 5000 degrees Kelvin and pressure 1 atmosphere) that lead to the formation of OOH<sup>•</sup>, OH<sup>•</sup> radicals. H<sup>•</sup>, O<sup>•</sup>. The phenomenon of cavitation consists of three phases: the formation of cavities by the creation of a vacuum due to negative pressure, the rapid growth of the cavities during the contraction and expansion of the waves and finally the collapse of the cavity and the formation of radicals. . The use of MgO nanoparticles as a catalyst in the presence of

ultrasonic waves can be considered as a method with higher efficiency. The process of pollutant removal is improved. The aim of this study was to evaluate the efficiency of the sonocatalytic process using magnesium oxide nanoparticles in removing cuticles from aqueous solutions.

## 2. Materials and methods

### 2.1. Materials and equipment

In this experimental study of phenolic compound with 100% purity from German Merck company, magnesium oxide nanoparticles from Zigma Aldrich, deionized water, hydrochloric acid and sodium  $MgCl_2 \cdot 6H_2O$  and NaOH hydroxide X1 and ElmasonicTI-H ultrasonic device with 130 kHz frequency A LUV-100A spectrophotometer with a wavelength of 285 nm oven and furnace were used.

To prepare magnesium oxide nanoparticles, 50 g of  $MgCl_2 \cdot 6H_2O$  was dissolved in 250 ml of distilled water to which 0 ml of 1-N NaOH solution had been previously added. The solution was stirred rapidly for 4 h to obtain the magnesium hydroxide precipitate. The suspension was then centrifuged. The resulting magnesium hydroxide precipitate was washed three times with distilled water and dried at 60 °C for 24 h. Finally, the dried powder was calcined in an oven at 450 °C for 2 h and MgO nanoparticles were obtained.

Conducting the experiments in a closed environment To study the effects of different parameters such as pH, initial concentration of catechol, dose of magnesium dioxide nanoparticles and contact time, a stock solution of the phenolic compound catechol was first prepared using double distilled water and solutions with concentrations (25, 50, 75., 100 and 150 mg/l.)

Then, in each phase of the experiment, 100 ml of the synthetic wastewater sample was added to a reactor with a diameter of 10 cm, a depth of 40 cm and a volume of 500 ml, and the pH was adjusted with hydrochloric acid and sodium hydroxide in the normal range (3, 5, 7 and 9.)

Different numbers of magnesium oxide nanoparticles (100, 150, 200, 400, 600, 800, 1000, 1200, 1600, 1800, and 2000 mg / l) were added to the sample and mixed with a magnetic stirrer.

After preparation, the sample was sonicated with 130-kHz ultrasonic waves in an ultrasonic device for a certain time (15, 30, 45, and 60 min)

Finally, the residual cuticle concentration in the samples was measured using the LUV-100A spectrophotometer at a wavelength of 285 nm and the cuticle removal efficiency was calculated using equation (1)

$$E = (C_0 - C_t) / C_0 \times 100 \quad (1)$$

Note that in all experiments three variables were kept constant and one variable was changed (optimisation method).

In this equation,  $C_0$  and  $C_t$ , in milligrams/litre, are the initial cuticle concentration and the concentration of catechol at time  $t$ , respectively.

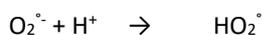
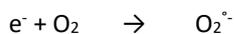
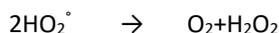
## 3. Results and discussion

### 3.1. Effect of variable

#### 3.1.1. The effect of pH on the efficiency of removal of catechol by the sonocatalytic process

The pH is one of the most important parameters in the removal of organic pollutants from wastewater, which greatly affects the adsorption capacity and structure of the pollutant, the distribution of electric charge on the catalyst surface, and the pathway and synthesis of the reaction material. (Saïen and Shahrezaei, 2012).

In this study, the results of the effects of pH on the removal of coke from aqueous solutions are shown in Figure 1. As is known, the optimum removal of coke occurred at pH 3, and with a further increase in pH, the removal efficiency gradually decreased, which was considered normal in this study. The removal efficiency for the initial concentration of 100 mg/l of catechol at this pH is 97.2%. The results of this study are in agreement with those of Bansal *et al.* (2015) and Peternel *et al.* (Peternel *et al.*, 2007). At acidic pH values, more hydroxyl radicals are formed in the solution due to the high concentration of  $H^+$  and sub-reactions. Therefore, the efficiency of organic pollutant removal increases at acidic pH values.



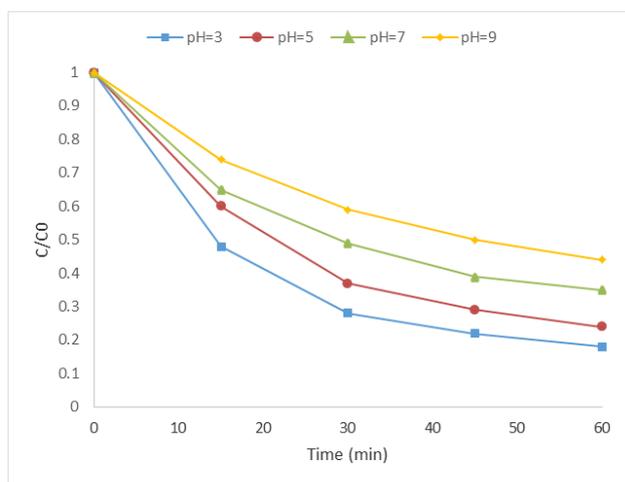
However, at high pH values,  $H_2O_2$  rapidly decomposes into  $H_2O$  and molecular oxygen, so that fewer hydroxyl radicals are formed and pollutant removal is less efficient (Kamani *et al.*, 2017). A study by Bazrafshan *et al.* on the removal of aniline with magnesium dioxide showed that the removal efficiency of aniline increased with increasing pH up to the neutral range. However, the removal of this pollutant decreases with increasing alkalinity (Bazrafshan *et al.*, 2016). Studies have also shown that the addition of nanoparticles gradually alkalises the environment, and when the pH is adjusted to the alkaline range, the environment becomes excessively alkaline, so under these conditions, the nanoparticles react with the water molecules and decompose, eventually losing their properties and decreasing the removal efficiency. (Sasaki *et al.*, 2011)

The effect of zinc oxide nanoparticle dose on the removal efficiency of catechol by sonocatalytic process

The dose of nanoparticles or adsorbents is one of the effective parameter in catalytic adsorption and oxidation processes (Bazrafshan *et al.*, 2014).

As shown in Figure 2, increasing the dose of magnesium oxide nanoparticles (100 to 2000 mg / l) increases the removal efficiency of catechol, so that more than 90% of the removal efficiency of catechol is achieved at a dose of

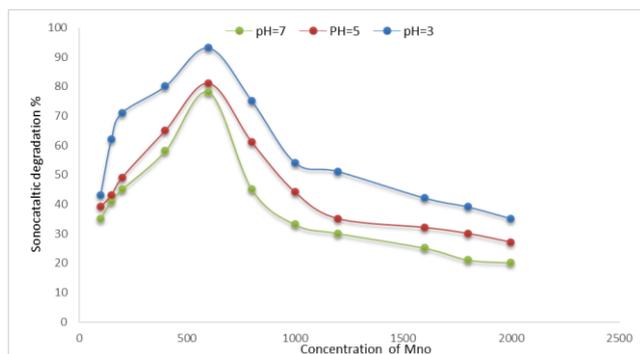
500 mg/l zinc oxide nanoparticles at a contact time of 60 min.



**Figure 1.** Effect of different pHs on removal of catechol using sonocatalytic process (MgO nanoparticle dose equal to 500 mg / L, initial catechol concentration 25 mg / L, frequency KH 130)

The results of the study by Fang *et al.* (2011), Dianti *et al.* (2014), Finance *et al.* (Daraei *et al.* (2014) are also in agreement with the results of this study.

At low concentrations of nanoparticles, the removal efficiency of pollutants decreases due to the long latency phase. Increasing the dose of MgO nanoparticles in the sonocatalytic process, in addition to shortening the latency phase, leads to the formation of nuclei and additional surfaces for cavitation. Enhanced (Haddadi *et al.*, 2007; Bazrafshan *et al.*, 2013) Increasing the concentration of nanoparticles makes more active sites available and increases the possibility of collision between organic contaminants and nanoparticles, but when their concentration exceeds a certain level, it acts as a radical scavenger and prevents the production and consumption of hydroxyl radicals. Shrinkage (Molinari *et al.*, 2006)



**Figure 2.** Effect of different doses of MgO nanoparticles on catechol removal using a sonocatalytic process: (initial concentration of catechol 25 mg / L, contact time 60 min and frequency KH 130)

Figure 2 describes the effect of contact time on the efficiency of cuticle removal by the sonocatalytic process. reaction time affects oxidation process design and performance, energy consumption and operating costs,

and describes the time to achieve the desired treatment. (Zazouli *et al.*, 2013) As can be seen, the number of impurities remaining in the solution gradually decreased by increasing the contact time from 15 to 60 min, and the maximum removal efficiency was reached after 60 min. The results of this study are in agreement with the results of the studies by Hosseini *et al.* (2013) and Kakavandi *et al.* (2014) Increasing the contact time at low concentrations of nanoparticles leads to increased production of free radicals and positive cavities, increases the cross-sectional area of adsorption and greater contact of catechol with free radicals, which increases the removal efficiency of the catechol during the sonocatalytic process Also, increasing the contact time by changing the active sites for adsorption of the cuticle and increasing the number of products resulting from the reaction of zinc in aqueous solution increases the removal efficiency of the catechol (Parastar *et al.*, 2012)

### 3.1.2. Kinetic studies

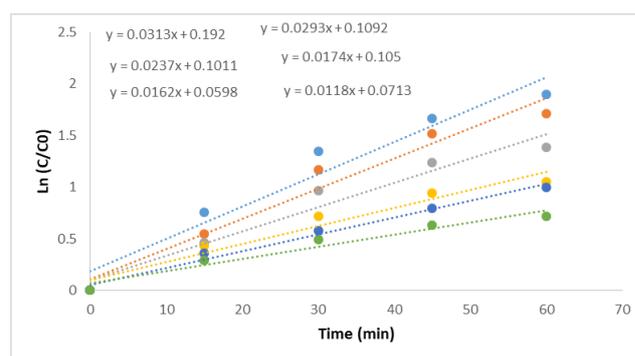
Sonocatalytic removal of the catechol using magnesium oxide nanoparticles follows a pseudo-first-order reaction. This kinetic was calculated according to the following equation

$$\ln \frac{C_0}{C} = K_{app} t \quad (2)$$

In this equation,  $C_0$  (mg / L) and  $C$  (mg / L) represent the initial concentration of the cuticle and the concentration at irradiation  $t$  (min), respectively.

According to Figure 3,  $K_{app}$  (1 / min) or in other words, the clear reaction rate constant was obtained by plotting the slope of the lines of Figure  $\ln (C_0 / C)$  versus  $t$ .

According to Figure 3, the  $K_{app}$  values related to sonocatalytic removal of the cuticle with increasing concentration from 25 mg/l to 200 mg/l were 0.0313, 0.293, 0.0237, 0.0174, 0.0162, and 0.0118, respectively



**Figure 3.** Kinetic diagram of  $\ln (C_0 / C)$  vs.  $t$ , in the removal of the catechol using the sonocatalytic process: (The dose of MgO nanoparticles is 500 mg / L, the initial concentration of the catechol is 25 mg / L and the frequency is 130 KH)

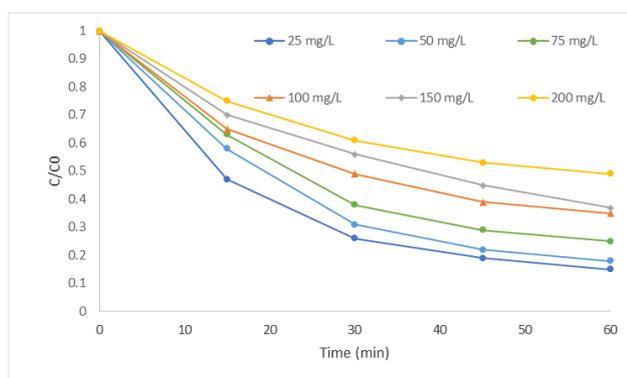
According to the correlation coefficient diagram in the presence of catalyst and at low concentrations, the cuticle is close to 1, which compared to higher concentrations, its values increased. (Khezrianjoo and Revanasiddappa, 2012) These results agree with studies by (Soltani and Safari, 2016) R. Darvishi Cheshmeh Soltani, M. Safari and Sahand

Jorfi *et al.* (2018) agreed that they used a sonocatalytic process using magnesium oxide nanoparticles with correlation coefficients ( $R^2 = 0.99$ ), respectively. And ( $R^2 = 0.951$ ). Obtained for pseudo-first degree kinetics.

### 3.1.3. The effect of catechol initial concentration on the efficiency of catechol removal by sonocatalytic process

Figure 4 shows that the removal efficiency of catechol decreased with an increase in its initial concentration, so that the maximum removal efficiency at the lowest initial concentration of catechol was 25 mg / L.

The results of this study are consistent with the results of the study by Elmolla *et al.* (2009) and Safari *et al.* (2014). As the concentration of contaminants increases, the concentration of oxidizing intermediates also increases, which increases the consumption of free radicals produced in the solution and reduces the removal efficiency of the catechol). However, by increasing the concentration of the catechol, the number of active sites available decreases due to more contaminant molecules adhering to the catalyst surface, thus reducing free radical production and removal efficiency (Kamani *et al.*, 2018). Samples with lower concentrations of contaminants decompose with the same number of free radicals than samples with high concentrations of contaminants (Jiang *et al.*, 2002). A study by Taherian *et al.* In 2013 showed that by increasing the concentration of p-chlorophenol from 10–200 mg/l, the degradation efficiency of this contaminant decreases during the sonocatalytic process of nanoparticles. (Taherian *et al.*, 2013)



**Figure 4.** Effect of different concentrations of catechol on the removal efficiency of sonocatalytic process: (pH 3, dose of MnO nanoparticles equal to 500 mg / L, contact time 60 min and frequency KH 130)

### 3.1.4. Langmuir-hinshelwood kinetics

The kinetics of oxidation and heterogeneous sonocatalytic degradation of catechol were studied by Langmuir-Hanschilwood model (Khuzwayo and Chirwa, 2015).

Typically, this kinetic model is used in the mechanisms of heterogeneous catalytic degradation of organic matter, the equation of which is expressed as follows (Shah *et al.*, 2015).

$$r = -\frac{dc}{dt} = K_r \theta_x = \frac{k_r KC}{1+KC} \quad (3)$$

In this equation  $r$ ,  $C$ ,  $t$ ,  $K_r$ ,  $k$  are the reaction rate (mg / min), the concentration of solution at any time (mg / l), the reaction time (min), the Langmuir Hanshillwood reaction rate mg mg/min and the Langmuir absorption constant, respectively. Are sonocatalytic constants of degradation (liters per milligram). The value of  $r$  according to the above formula was obtained at a concentration of 25 mg/l 0.84851.

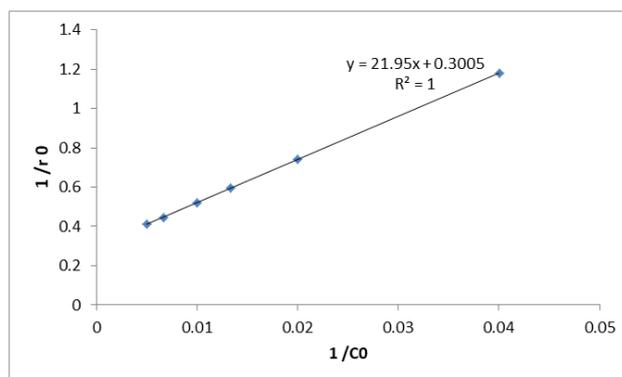
Additionally, the kinematic parameters of Langmuir-Hanschilwood model

Can be calculated based on the following linear equation.

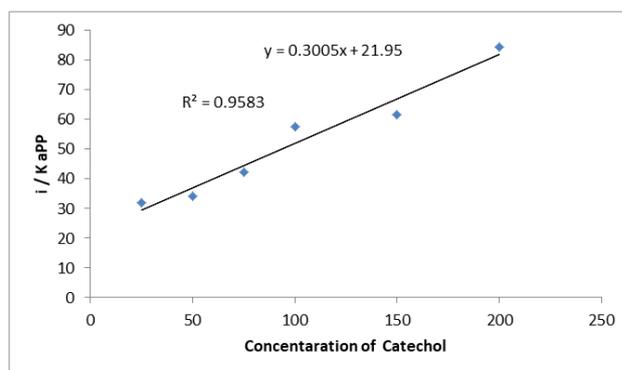
$$\frac{1}{r_0} = \frac{1}{k_r} + \frac{1}{k_r k C_0} \quad (4)$$

According to Figure 5, the numerical values of  $K$  and  $K_r$  at an initial concentration of 25 mg / l were 0.1369 and 3.3272, respectively.

To determine the linear synthetic model, pH of  $1/k_{app}$  versus  $1/c_0$  was plotted. As shown in the Figure, an almost linear relationship with  $R^2$  (0.9583) was obtained, which shows. According to Figures 6 and 7, the concentration of catechol versus  $1/k_{app}$  and the catalytic degradation of catechol follow the Langmuir-Hanschilwood kinetics (Images 1 and 2).

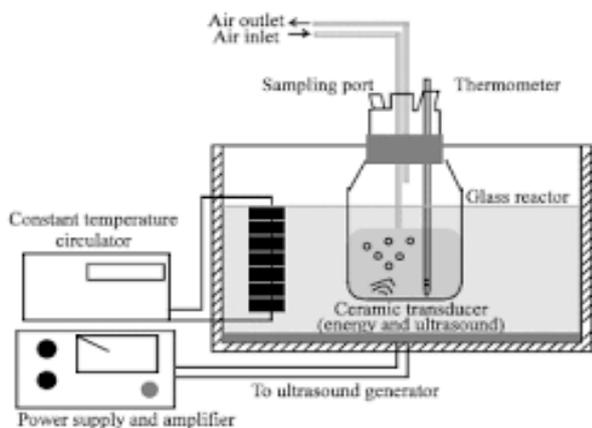


**Figure 5.** Kinematic studies of the Langmuir-Hanschilwood model based on the reciprocal variance of  $1/r_0$  vs.  $1/c_0$

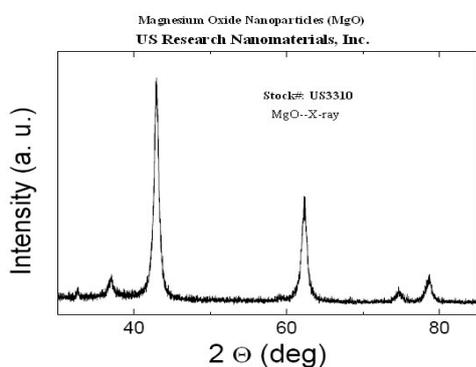


**Figure 6.** Kinetic studies of the Langmuir-Hanschilwood model based on the reciprocal variance of  $1/k_{app}$  against initial catechol concentrations

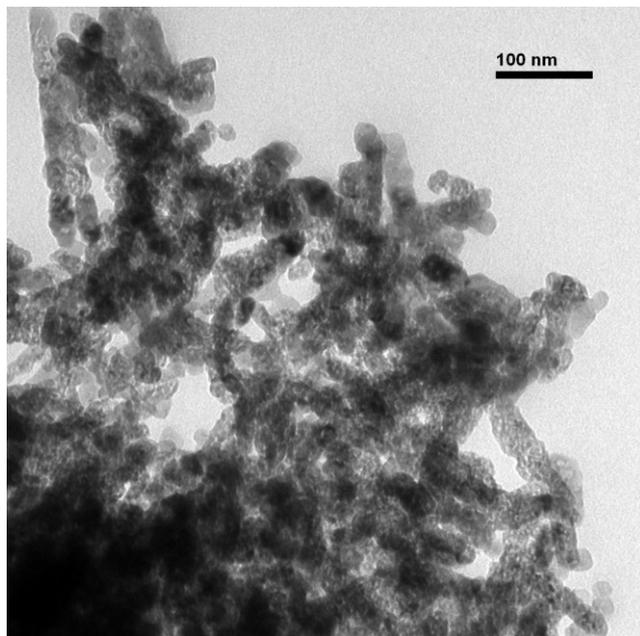
### 3.2. Study of Characterization



**Image 1.** Image of a sonocatalyst reactor.



**Figure 7.** Three peaks appear at angles of 42, 62, and 80  $2\theta$ , which, when compared with other synthesized non-particles, indicate the proper synthesis of MgO



**Image 2.** Investigation of the average size of nanoparticles  
Investigation of the surface morphology of nanoparticles

## 4. Conclusion

This study evaluates the efficiency of sonocatalytic process using magnesium oxide nanoparticles in the removal of

catechols from aqueous solutions in the values of effective parameters, including pH equal to 3, the dose of MgO nanoparticles equal to 500 mg/l, initial concentration of 25 mg/L catechol at 130 KH frequency.

The maximum degradation efficiency was obtained by increasing the nanoparticle dose and decreasing the initial concentration of catechol. Also, considering the values of R2, which is close to one, the kinetics of this study follows the pseudo-first-order reaction and the kinematic model of Langmuir Hanschlwood;

Therefore, the sonocatalytic degradation process performed by magnesium oxide nanoparticles to remove catechols from aqueous solutions is an easy, practical and cost-effective method that can be used to degrade various organic materials due to their specific properties. Other particles can be used as catalysts in the removal of catechol.

### Ethical Approval

Code of Ethics approval is not required because no human or animal studies have been conducted.

### Conflict of interest

The authors declare have no Conflict of interest

### Author contribution

H K; idea of the study+H A; writing+M H; experiments +A H; analyzes +A M; experiments

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