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Metal-Free Activated Carbon Catalytic for Degradation of Organic Contaminants by Peroxydisulfate Activation

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ABSTRACT: Green chemistry has recently become trending, and the discovery of environmentally friendly catalysts is mandatory. Activated carbons (ACs) are one of the most environmentally friendly yet cheap materials with the potential for catalyst application. Three commercially available ACs from Pancasari, Norit, and Chemical Supply were used as metal-free catalysts for advanced oxidation process (AOP) phenol removal in water in the presence of oxidants peroxydisulfate (PDS), and hydrogen peroxide (H_2O_2). It was found that ACs were effective in being used as catalysts for activating those oxidants to oxidize phenol in AOP reactions. In this study, the surface area of the catalyst significantly improved the phenol removal efficiency. ACs Pancasari (ACP) with the highest surface area has the best degradation performance of phenol removal with up to 99% removal efficiency in 60 minutes under the condition of [phenol] = 30 mg L^{-1}, [ACP] = 0.2 g L^{-1}, [PDS] = 3.3 mmol L^{-1}, and T = 25°C. it was also found that the degradation process was significantly influenced by reaction temperature. Nevertheless, in this study, ACs display the potential as catalysts in the AOP process for wastewater treatments.

Key words: Green catalyst, wastewater, AOP, activated carbon, peroxydisulfate

1. INTRODUCTION

As a pollutant, phenol can be found in the water from industrial discharge since it is used as a precursor in many industrial processes and reactions. It causes harm to the environment in high concentrations since it has toxic properties to humans and animals in general [1]. Also, phenol has very stable chemical properties due to its aromatic structure and highly dense electrons available in the benzene ring or the hydroxyl group, making it hard to degrade in natural process [2, 3].

Considering the stable chemical properties of the phenol, Advanced Oxidation Process (AOP) degradation becomes the alternative for phenol removal in the wastewater. Sulfate ($SO_4^{\bullet -}$) radical generated from peroxymonosulfate and/or peroxodisulfate (PMS/PDS) using a catalyst process is commonly studied in the extent of application of the degradation process [4]. Oxidant persulfate, in this case, PDS ($S_2O_8^{2-}$) can be activated using a catalyst to produce either $SO_4^{\bullet -}$ and/or $^1O_2$ [5]. $SO_4^{\bullet -}$ radicals have various advantages, namely $SO_4^{\bullet -}$ radicals having higher standard reduction potential (E_0 = 2.5-3.1 eV) compared to $OH^{\bullet}$ radicals (E_0 = 1.8-2.7 eV) [6]. The higher standard reduction potential means $SO_4^{\bullet -}$ radicals are highly reactive to the degradation of organic pollutants. Moreover, $SO_4^{\bullet -}$ has a longer half-life, meaning it is stable at the reaction time. $SO_4^{\bullet -}$ radicals can be produced in a wide pH range. Thus, they can be used in either acidic or alkaline conditions [7]. Many studies also showed that $SO_4^{\bullet -}$ based oxidation outperforms $OH^{\bullet}$ radicals in terms of total organic carbon (TOC) removal and mineralization to CO_2 and H_2O of organic pollutants [8, 9]. Therefore, applying the sulfate radical advanced oxidation process (SR-AOP) in phenol degradation is very beneficial to this study. Various catalysts have been studied in favor of generating $SO_4^{\bullet -}$ radical in the process. In previous studies, transition metal oxide-based catalysts such as Mn, Co, Fe, Ag, etc., such as Mn, Co, Fe, Ag, etc., have been extensively used [10-13]. However, recent studies have been conducted to use a non-metal-based catalyst to minimize the metal toxicity discarded in the environment after the wastewater treatment process. Carbonaceous materials such as graphene and carbon graphite nitride were investigated as alternative catalysts for producing $SO_4^{\bullet -}$ radicals [14, 15]. However, using those materials has economic hindrances since graphene and carbon graphite nitride are relatively hard to produce and rather expensive. In this paper, other alternative materials, such as commercially available activated carbon (AC) powder, were studied. With the properties of AC, such as huge surface area, porous structure, environmental friendliness, and economic viability, AC is the perfect

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candidate as a catalyst in the AOP water treatment reaction [16, 17]. The reactiveness of the AC surface to promote $SO_4^{\bullet-}$ radical was extensively studied in favor of degrading organic pollutants such as phenol as in this study. It happened because of the properties of carbon configuration, oxygen functional group, surface defects, and dimensional structure [6, 18].

In this paper, we report the performance of commercial activated carbons that are based on low-cost materials, namely AC Pancasari Puspa (ACP), AC Norit (ACN), and AC Chemical Supply (ACC), as catalysts for phenol removal. Their physicochemical properties, reaction kinetics, catalytic activities, and regeneration of used catalysts were also investigated.

2. EXPERIMENTAL SECTION

Three commercially available activated carbons, namely AC Pancasari Puspa (ACP), AC Norite (ACN), and AC Chemical Supplies (ACC) were obtained from a local chemistry store. The catalysts are used without any further treatment. The Peroxydisulfate as oxidant (PDS) and H_2O_2 (30 wt%), phenol (99% purity), and methanol (Merck, 99% purity) were supplied by Merck-Germany.

The AC surface area was measured using N_2 adsorption-desorption using autosorb-1 quantachrome (USA). The catalyst samples were degassed at 100°C for 24 h. The surface area was then calculated using Braunauer-Emmet-Teller (BET) methods. Thermogravimetric analysis (TGA) was conducted in argon at a heating rate of 10°C/min on a Perkin-Elmer Diamond TG/DTA thermal analyzer.

Catalytic oxidation of phenol was carried out in a 1 L reactor with a temperature controller containing 500 mL of phenol at different concentrations. A fixed amount of PDS as a radical source and catalyst was added and entirely dissolved before the reaction was started. The reaction was done by stirring the mixture at 400 rpm. The phenol concentration was analyzed using HPLC with a UV detector at a wavelength of 270 nm. The column used was C-18 with a mobile phase of 30% CH_3CN and 70% H_2O. Using an activated carbon catalyst, H2O2 was also employed to obtain different oxidant effects in phenol degradation. A fixed amount of H_2O_2 was added to the degradation reaction. Temperature effect SR-AOP data was obtained using each catalyst ACP, ACN, and ACC at three different controlled temperatures 25 °C; 30 °C; and 35 °C. Then, the phenol concentrations were analyzed using HPLC with a UV detector. First, the catalysts obtained reusability data and were washed with ethanol and water before each cycle to remove the substrate from the catalyst's surface. Then, each catalyst was used for a reusability test. After that, the phenol concentration was analyzed using HPLC with a UV detector. To understand the reaction rate of the catalysts, the kinetic of phenol degradation was investigated using pseudo-first-order models using the equation as follows [19]:

$$\mathrm{Ln}\ \left({}^{Ct}/_{Co}\right) = -k_{obs} \bullet t \qquad (1)$$

Where k_{obs} are the observed (obs) rate constant, t is time, C_0 is the initial phenol concentration, and C_t at the time (t). To obtain the activation energy of all the AC catalysts, a linear Arrhenius equation was used:

$$\mathrm{Ln}(k) = {}^{Ea}/_{RT} + A \qquad (2)$$

3. RESULT AND DISCUSSION

3.1 Cool Down Test

The surface properties of the AC materials, the N_2 sorption isotherms of ACP, ACN, and ACC are shown in Fig. 1 below. Moreover, the surface area and pore radius of ACP, ACN, and ACC catalysts are presented in Table 1.

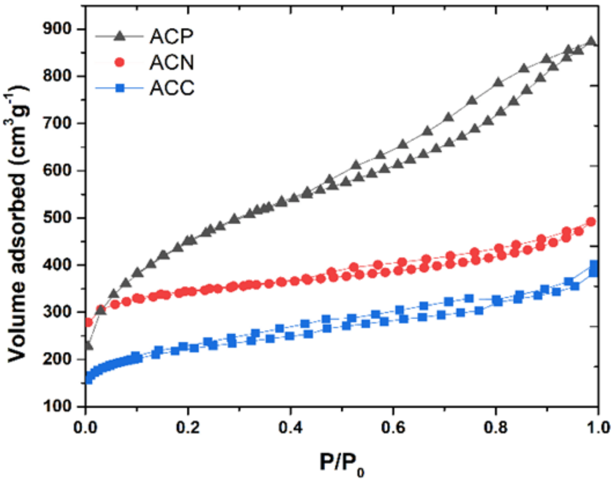

Fig. 1. N_2 adsorption-desorption isothermal of carbon catalysts

The BET N_2 isotherm sorption properties of the carbon active catalysts, as shown in Fig. 1, show the type 4 isotherms with H_4 type hysteresis. This means all of the catalysts have mesoporous pore types. The carbon ACP materials have exceptional surface area compared to ACN and ACP carbon (860 vs. 586.1 vs. 360.9 $m^2\ g^{-1}$) with mesoporous pore properties (22.1, 24.2, and 16.9 Å). The high surface area in the catalyst theoretically enhances the mass transfer between the substrate and oxidant-radical on the surface and active sites of the catalyst.

Table 1 Surface properties analysis of ACP, ACN, and ACC catalyst

Catalyst	S_{BET} ($m^2 g^{-1}$)	Pore radius (Å)
ACP	860.01	22.12
ACN	586.11	24.24
ACC	360.90	26.45

The result of the thermographic analysis (TGA) of each activated carbon is presented in Fig. 2. For the ACP catalyst, there is a slight mass reduction of around 10% at 100°C, which can be ascribed as water loss from the surface of the ACP materials. However, in the other two materials, it doesn't occur. This means ACP has more water content than other materials, namely ACN and ACC. However, all materials materials have a significant loss mass of around 80% at 450-600°C [20]. It can be attributed to the loss of acidic and oxygen functional groups by thermal processes for each AC material at those temperatures.

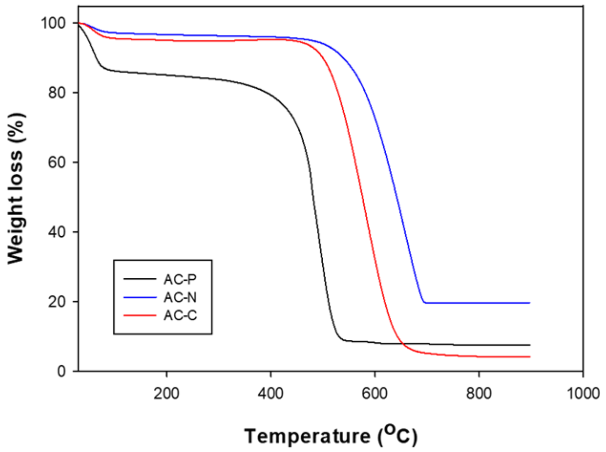

Fig. 2. Thermographic analysis of each AC material

3.2 Preliminary Study

Fig. 3 below presents the performance of catalysts for phenol removal. To understand the removal reaction processes, ACP, ACN, and ACC catalysts were used without the presence of the oxidant. The same reaction control for all catalysts was used to determine the best effectivity in phenol removal degradation.

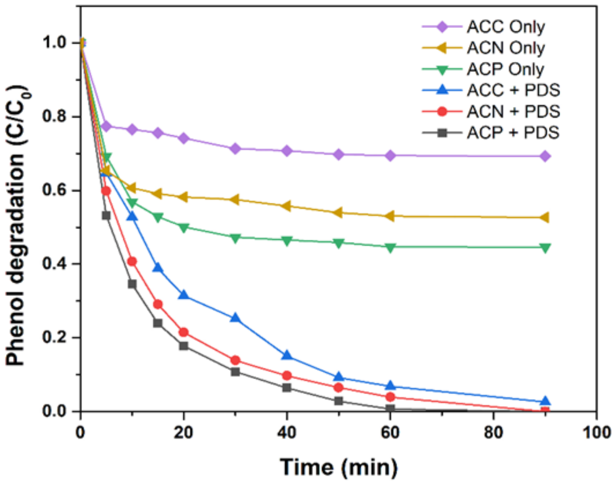

Fig. 3. Comparison of the performance of AC catalysts for Phenol removal. Conditions of reaction: C_0 = 30 mg/L; [PDS] = 3.3 mmol/L; catalyst dosage 0.05 g/L; temp = 25°C.

Firstly, AC, with exceptionally high surface area, has somewhat good adsorption performance. As shown in Fig. 2, ACP has 50 % of phenol adsorption capacity. ACN and ACP have 42% and 23% phenol adsorption capacity, respectively. This can be ascribed to ACP having the largest surface area. Furthermore, in the oxidation study after the addition of PDS, ACP also had a better overall performance with almost 100% phenol removal efficiency compared to the other catalysts, which had 99 % and 96 % phenol removal. The reaction kinetics results for each catalyst are presented in Table 2 below.

As shown in Table 1, ACP has the highest reaction rates in phenol removal compared to the ACP and ACC catalysts. This study shows that carbonaceous catalysts with larger surface areas generally have better degradation efficiency. A higher surface area, meaning the mass transfer process between oxidant and phenol components, could be adequate for the catalyst's active site [21, 22]. In addition, the higher surface area also means more active sites are available on the surface of the catalysts [23]. The reaction of degradation itself started when PDS was activated on the surface of the AC catalyst to produce radicals as it follows the reactions [24]:

Table 2 The rate constant (k_{obs}) of the AC catalysts

Catalyst	k_{obs} (min^{-1})
ACP	0.13
ACN	0.08
ACC	0.04

$$AC + S_2O_8^{2-} \rightarrow AC^{+} + SO_4^{\bullet-} + SO_4^{2-} \quad (3)$$
$$SO_4^{\bullet-} + H_2O \rightarrow H^{+} + SO_4^{2-} + OH^{\bullet} \quad (4)$$
$$SO_4^{\bullet-} + OH^{-} \rightarrow SO_4^{2-} + OH^{\bullet} \quad (5)$$
$$SO_4^{\bullet-} + OH^{\bullet} + C_6H_5OH \rightarrow CO_2 + H_2O \quad (6)$$

As can be seen in the reaction above, $SO_4^{\bullet-}$ could react with water to produce $OH^{\bullet}$. Hereinafter, either of the radicals ($OH^{\bullet}$ and $SO_4^{\bullet-}$) could react with phenol during the degradation process.

3.3 Temperature Effect

Temperature, in general, is one of the most significant factors in the organic reaction process. Fig. 4 below shows the temperature influence of the phenol in all of the AC catalysts.

The figures above show that the higher temperature is followed by the increased phenol degradation rate. For instance, all three catalysts have almost 100% phenol removal efficiency at 45°C and get less removal efficiency at lower temperatures. With the results of the temperature-dependent reaction, the phenol removal reaction using AC catalyst is an endothermic reaction [19, 25].

The calculated activation energy of each catalyst is displayed in Table 3 below.

Table 3 The activation energy of the AC catalyst

Catalyst	Ea (kJ mol^{-1})
ACP	24.51
ACN	35.14
ACC	37.29

From the data above, ACP has the lowest activation energy, 24.51 kJ mol^{-1} followed by the ACN 35.14 kJ mol^{-1} and ACC 37.29 kJ mol^{-1}. Those catalysts have relatively

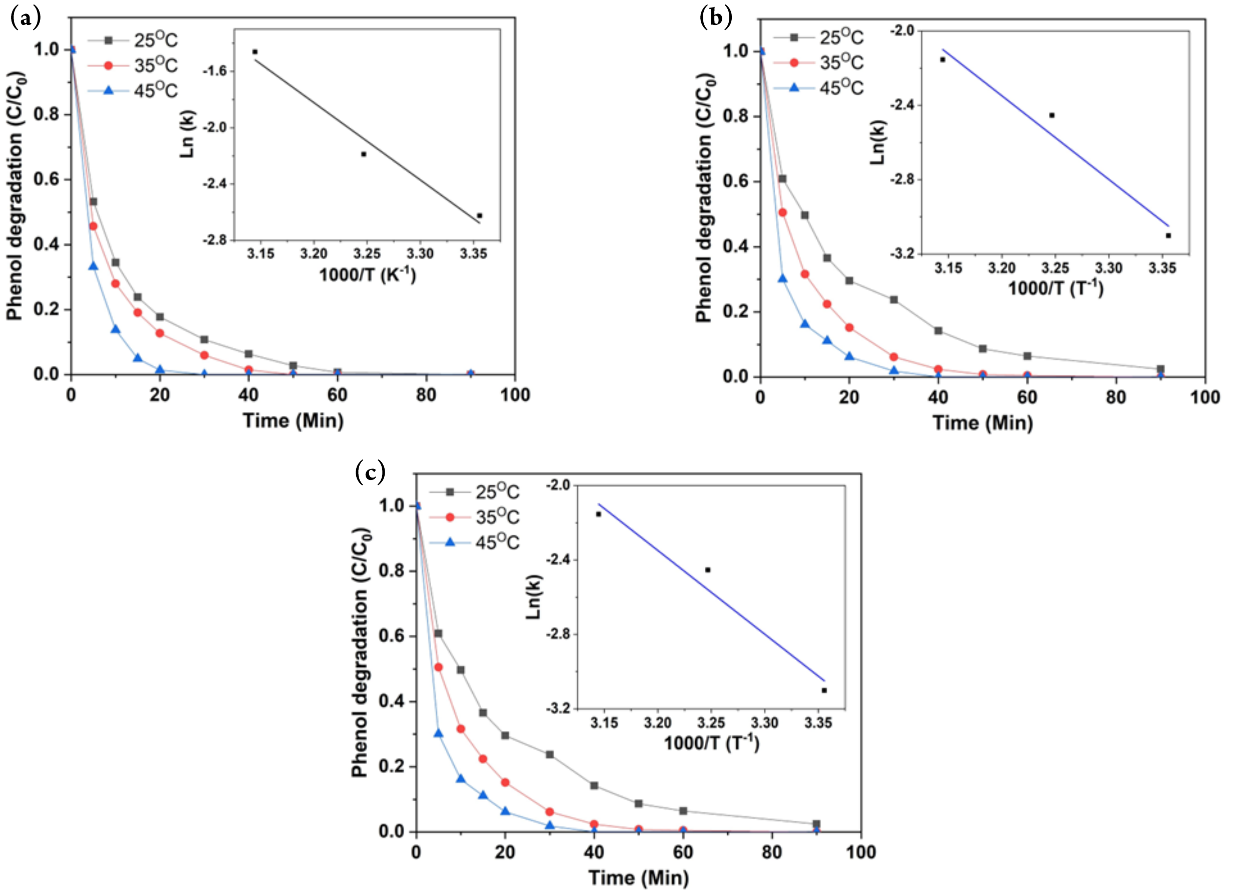

Fig. 4. Effect of temperature on phenol removal on three different catalysts. ACP (a), ACN (b), and ACC (c). Conditions of reaction: $C_0 = 30$ mg L^{-1}; [PDS] = 3.3 mmol L^{-1}; catalyst dosage 0.05 g L^{-1}.

lower activation energy than inorganic AOP catalysts such as Co/AC, RuO_2/AC, cobalt-based, and Fe_2O_3 [26].

3.4 Different Oxidant Effects

The effect of different oxidants on phenol degradation was also studied using PDS and H_2O_2 with ACP as the catalyst. The result is shown in Fig 5 below.

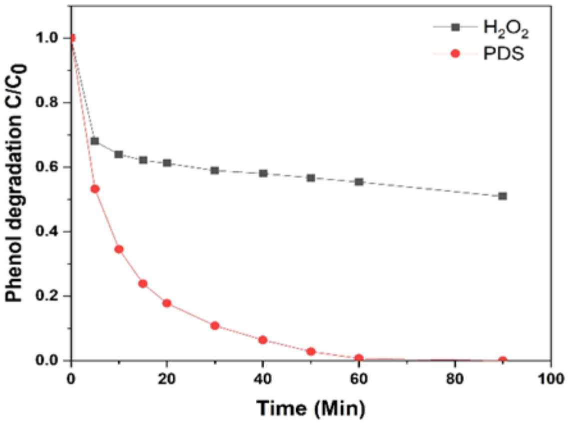

Fig. 5. Different oxidants on phenolic degradation using ACP catalyst. Conditions of reaction: $C_0 = 30$ mg L^{-1}; [Oxidants] = 3.3 mmol L^{-1}; catalyst dosage 0.05 g L^{-1}; temp = 25°C.

As can be seen, PDS oxidant has better degradation efficiency compared to H_2O_2 oxidant, almost 100 % vs. 51 %, with k_{obs} of 0.13 min^{-1} vs. 0.0007 min^{-1}. Previous studies showed that carbon surface could activate H_2O_2 to form $OH^\bullet$. Moreno Castilla et al. [27] suggest that the active surface of the AC has delocalized π electrons, which could be transferred to oxidize H_2O_2 and produce $OH^\bullet$ radicals the reactions as follows:

$$C\text{-}\pi + H_2O_2 \rightarrow C\text{-}\pi^+ + OH^- + OH^\bullet \quad (7)$$

$$C\text{-}\pi^+ + H_2O_2 \rightarrow C\text{-}\pi + H^+ + O_2H^\bullet \quad (8)$$

However, $OH^\bullet$ is less reactive compared to $SO_4^{\bullet-}$ radicals since it has lower oxidation potential compared to $SO_4^{\bullet-}$ (1.8 – 2.7 eV vs. 2.5 – 3.1 eV). Also, $SO_4^{\bullet-}$ could oxidize water to produce $OH^\bullet$, which means in the PDS system, $OH^\bullet$ and $SO_4^{\bullet-}$ radicals are available in the degradation process. Therefore, the degradation process is more efficient when this study uses PDS as an oxidant [28].

3.5 Mechanism Reactions

The mechanism could be predicted from the HPLC results in Fig 6. The degradation product is used as an ACP catalyst at the initial, 5, and 30 minutes after reaction.

The initial state of HPLC showed three peaks of methanol, water as the solvent, and phenol with retention times of 7, 10, and 17 min, respectively. After 5 minutes, the reaction occurred, and new peaks appeared. Those peaks were 4-hydroxybenzoic acid, benzoquinone, and hydroquinone, while phenol peaks were subsidized. Furthermore, after 30 minutes of reaction, the phenol peak was disappeared. The predicted product of SR-AOP degradation is shown in Fig 6. Degradation started with the phenol oxidation to 4-hydroxybenzoic acid in the presence of oxygen and sulfate radicals from PDS occur in para position [29]. The remaining 4-hydroxybenzoic acid is then oxidized into hydroquinone by sulfate radicals. The sulfate radicals will directly react with 4-hydroxybenzoic acid to form intermediate aromatic cationic radicals [30]. The presence of water and/or hydroxy radicals will produce a hydration reaction to form hydroquinone. Hydroquinone is then easily oxidized into benzoquinone [31].

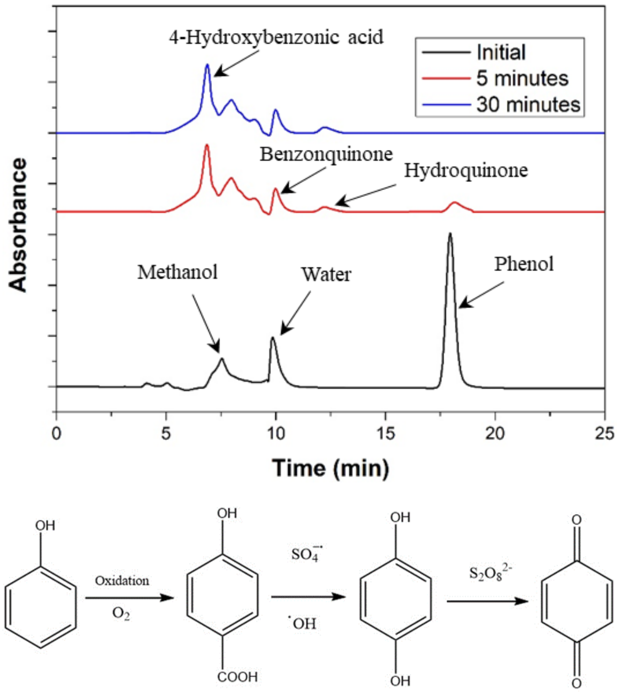

Fig. 6. HPLC chromatogram of Phenol degradation and intermediate product of AC catalyst

3.6 Catalyst Reusability

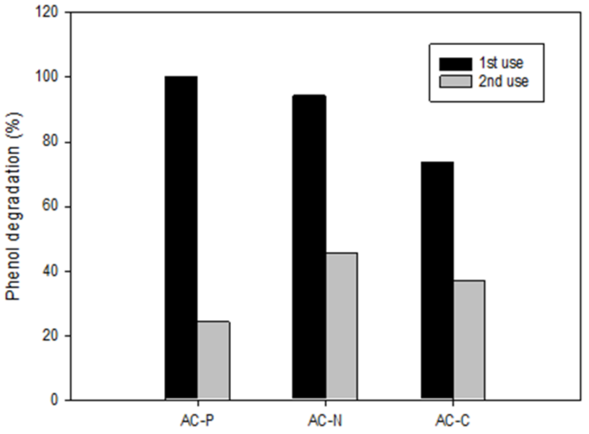

Fig. 7. Reusability of AC-P, AC-N, and AC-C catalyst in AOP reactions. Conditions of reaction: C_0 = 30 mg/L; [PDS] = 3.3 mmol/L; catalyst dosage 0.05 g/L; temp = 25°C.

The stability of the catalyst was obtained by re-using each catalyst. Catalysts were washed with ethanol and water before each cycle to remove the substrate from the catalyst's surface. After the second usage of the catalyst, the removal efficiency was dropped to below 50% for each catalyst, as shown in Fig. 7 above. In a study conducted by Silambarasan, carbon-based catalysts tend to have a strong affinity toward organic substances of oxidized products, which obstructs the active sites of the catalyst and reduces the catalyst's effectivity [32].

4. CONCLUSION

AC could be one of the alternative solutions to treat wastewater problems in the future since, compared to other catalysts, AC is relatively cheap and environmentally friendly. As from this study, three commercial ACs, namely activated carbon Pancasari (ACP), activated carbon Norit (ACN), and activated carbon Chemical Supply (ACC) could be used as a catalyst for the SR-AOP process in phenol removal applications. ACP, with a higher surface area than the other two, performs better phenol degradation. The degradation process in this study follows the first-order kinetics, and the activation energy of these catalysts is obtained as 24.51, 35.14, and 37.29 kJ mol^{-1} for ACP, ACN, and ACC, respectively. ACP, in this study, could activate both PDS and H_2O_2 to produce $SO_4^{\bullet-}$ and $OH^{\bullet}$ radicals. Yet, the best efficiency is obtained using PDS as an oxidant source.

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