

# Synergetic Effect of TiO<sub>2</sub>–PCC/Zeolite Nanocomposite on the Photodegradation of Phenolic Compound in Wastewater

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**Abstract.** Phenol is an organic compound commonly found in the water bodies contained polluted wastewater from industrial, agricultural, and domestic activities. This compound exhibits carcinogenic properties and can impact human health at certain concentrations. Therefore, excessive phenol must be degraded. Various techniques dealing with phenol waste have been developed, including the adsorption method. However, secondary pollutants might form after the adsorption process. One potential alternative for handling phenolic compounds in wastewater without generating secondary waste is the photocatalytic process. It has been proven that, when combined with adsorption, the degradation activity can be enhanced. In this paper, phenol degradation was investigated by carrying out degradation using a semiconductor catalyst, such as TiO<sub>2</sub>, that synergizes with adsorbents like precipitated calcium carbonate (PCC) and zeolite. The highest phenol degradation was achieved with the variables 80% TiO<sub>2</sub> - 20% PCC and 80% TiO<sub>2</sub> - 20% zeolite, resulting in 74.3% and 69.22% of phenol degradation, respectively. In comparison, the TiO<sub>2</sub>–PCC nanocomposite exhibits superior performance to the TiO<sub>2</sub>–zeolite nanocomposite. The observation of the functional groups revealed that PCC has a larger functional group area, supporting the synergy of the adsorption-photocatalysis process compared to the TiO<sub>2</sub>–zeolite nanocomposite.

**Keywords:** Advanced Oxidation Processes, Photocatalysis, Phenol, Zeolite, PCC

## INTRODUCTION

Phenol is an organic compound derived from industrial, agricultural, and domestic waste, which is both harmful to the environment and humans. In certain concentrations, these compounds can have

adverse effects on humans, including liver and kidney damage, decreased blood pressure, weakened heart rate, and even death (Bibi *et al.*, 2023; Cui *et al.*, 2023; Viet *et al.*, 2023). According to Regulation No. 6/2021 of the Minister of Environment and Forestry of the Republic of Indonesia, the

maximum quality standard for phenol in wastewater is 0.5 mg/L, and the threshold for phenol in drinking water is 0.002 mg/L, as stated by the Environmental Impact Control Agency (Tanjung *et al.*, 2022). Therefore, phenol must be degraded from wastewater before discharge (Wardhani & Dirgawati, 2011).

Currently, various techniques or methods for dealing with phenol waste have been developed and applied, one of which is the adsorption method (Bibi *et al.*, 2023; Cui *et al.*, 2023; Sharafinia *et al.*, 2023). However, this method proved to be less effective because the adsorbed phenol still accumulates in the adsorbent, leading to the generation of secondary pollutants. Other conventional water treatment technologies, such as sedimentation, filtration, chemical use, and membrane technology, incur high operating costs and, similarly, can generate toxic secondary pollutants (Bibi *et al.*, 2023).

One potential alternative for handling phenolic compounds in waste is the photocatalytic process. The photocatalytic method is relatively energy-efficient because it can utilize sunlight and degrade pollutants into non-toxic compounds (Viet *et al.*, 2023). The photodegradation method can be carried out using a semiconductor catalyst. TiO<sub>2</sub>, ZnO, CdS, and Fe<sub>2</sub>O<sub>3</sub> are the most frequently utilized semiconductor catalysts (Permata *et al.*, 2016). TiO<sub>2</sub> is the most effective semiconductor catalyst because it is non-toxic, affordable, and naturally abundant. Modifications to the structure, surface area, and particle size of TiO<sub>2</sub> may enhance its photocatalytic activity (Ariyanti *et al.*, 2018). One method is to combine TiO<sub>2</sub> with the adsorbent (Syafii, 2019). Precipitated calcium carbonate (PCC) is an adsorbent that exhibits properties comparable to TiO<sub>2</sub>, particularly in photocatalytic applications.

Similarly, zeolite is a porous material known for its ion exchange, adsorption, and catalytic capabilities. Both PCC and zeolite represent promising candidates for the development of TiO<sub>2</sub>-based nanocomposite materials with enhanced photocatalytic performance. These materials are also abundantly available, further supporting their potential for large-scale application.

This paper reports on the synthesis of PCC-TiO<sub>2</sub> and Zeolite-TiO<sub>2</sub> nanocomposites, their characteristics, and their application for phenol degradation. The nanocomposite is expected to serve as an alternative to the hospital waste treatment process, reducing risks to the environment and organisms, being efficient in terms of economics and energy use, and not producing secondary waste.

## MATERIALS AND METHODS

### Materials

TiO<sub>2</sub> nano-powder with a size <25 nm was used as a nanocomposite raw material. TiO<sub>2</sub> nano-powder, tetraethyl orthosilicate (TEOS), and precipitated calcium carbonate (PCC) were purchased from Sigma-Aldrich Co. LLC and used without further purification. Meanwhile, natural zeolite obtained from Gunung Kidul, Yogyakarta (Indonesia) was treated and activated before use. The steps were as follows: grain size reduction, chemical activation with 0.5 N hydrochloric acid (HCl) for 80 minutes while stirring, and neutralization with distilled water. Finally, calcination was performed at 300°C for 2 hours.

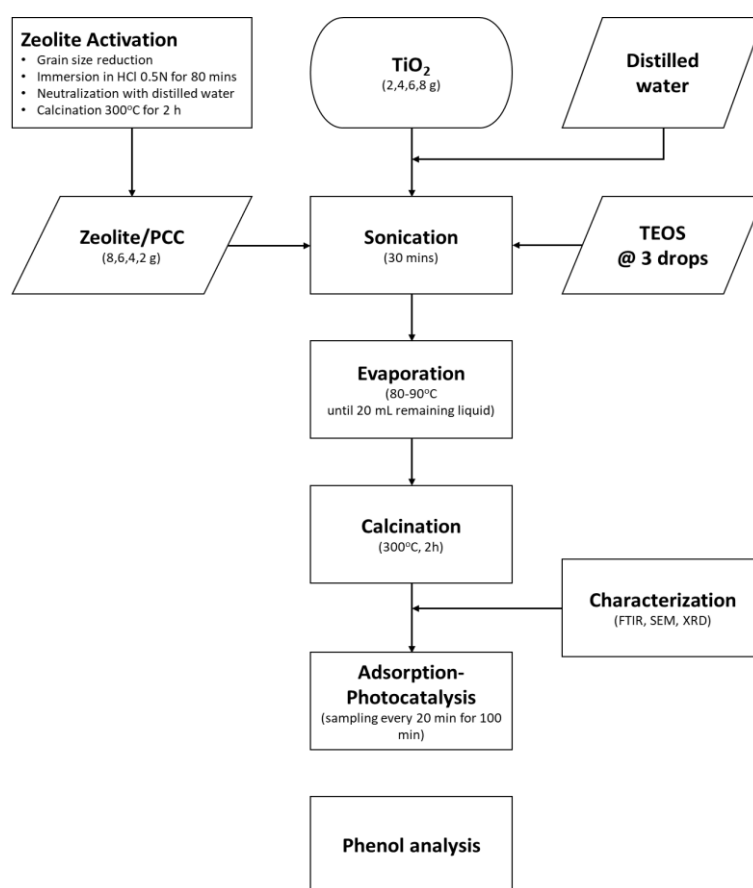
### Synthesis of TiO<sub>2</sub>-PCC and TiO<sub>2</sub>-Zeolite Nanocomposites

Nanocomposite with a total basis of 10 g consisting of TiO<sub>2</sub>, which was weighed

separately with the ratios (20%, 40%, 60%, 80%). First,  $\text{TiO}_2$  was dissolved in 100 mL of distilled water and then sonicated for 30 minutes. After sonication, 3 drops of TEOS were added, and the sample was sonicated again for 2 minutes. Then PCC or zeolite was added in a weight percent ratio (80%, 60%, 40%, 20%), followed by stirring and sonication for 30 minutes. The procedure then continues with heat treatment at  $80^\circ\text{C}$  to  $90^\circ\text{C}$  with continuous stirring until the liquid volume reaches approximately 20 mL to 30 mL. The liquid is then calcinated in the furnace at  $300^\circ\text{C}$  for 2 hours. The sequence of  $\text{TiO}_2$  nanocomposite synthesis is shown in Figure 1, and the experimental framework is presented in Table 1.

**Table 1.** Variable of  $\text{TiO}_2$  nanocomposite

Run	$\text{TiO}_2$ (g)	PCC / Zeolite (g)	Sampling time (min)
1	2	8	20
2			40
3			60
4			80
5	4	6	20
6			40
7			60
8			80
9	6	4	20
10			40
11			60
12			80
13	8	2	20
14			40
15			60
16			80



**Fig. 1:** Flowchart of the synthesis of  $\text{TiO}_2$  nanocomposites

### Nanocomposite Characterizations

All samples of TiO<sub>2</sub>-PCC and TiO<sub>2</sub>-Zeolite nanocomposites were analyzed using scanning electron microscopy-energy-dispersive X-ray or SEM-EDX (JEOL JSM-6510LA) to determine its surface morphology, Fourier transform infrared spectroscopy (FTIR) using Perkin-Elmer UATR Spectrum Two to determine the chemical bonds and functional groups of the nanocomposites, and X-ray diffraction (XRD) using SHIMADZU XRD-7000 to analyze the structure of the crystalline material of the nanocomposite.

### Degradation of Phenol by Adsorption-Photocatalytic Method

Approximately 3 g of the nanocomposite was added to 200 mL of a 2 g/L phenol solution and then transferred into the photocatalytic reactor for the degradation process, assisted by black lamp light (400 nm).

Sampling was carried out every 20 minutes to 80 minutes, with a volume of up to 20 ml, for phenol concentration measurement using a Vis Spectrophotometer.

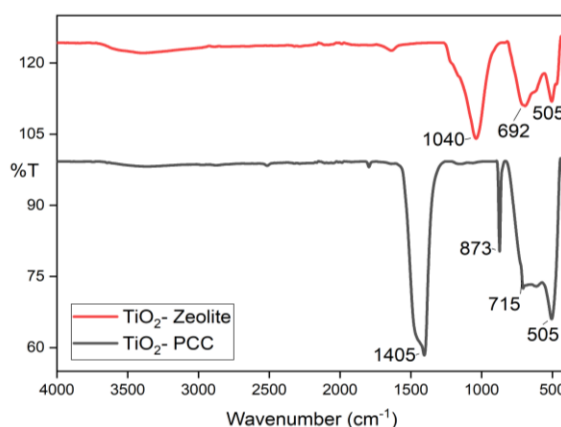
## RESULTS AND DISCUSSION

### Nanocomposite Characterization

#### Fourier Transform Infra-Red (FTIR)

FTIR analysis was performed to identify the presence of functional groups in the nanocomposite. According to Figure 2, the functional groups on the TiO<sub>2</sub>-PCC photocatalyst, as determined by FTIR, exhibit peaks at wavelengths of 1405 cm<sup>-1</sup>, 873 cm<sup>-1</sup>, 715 cm<sup>-1</sup>, and 505 cm<sup>-1</sup> with varying intensities. The peak areas at 1405 cm<sup>-1</sup> are attributed to the bending vibration of the O-H group from a water molecule or Ti-OH (Yu *et al.*, 2003). The peak at a wavelength of

873 cm<sup>-1</sup> indicates an out-of-plane bending vibration of the carbonate ion in calcite. In contrast, the peak at 715 cm<sup>-1</sup>, with a low intensity, indicates an in-plane bending vibration of the carbonate ion in calcite (Somarathna *et al.*, 2016). Calcite is the crystalline form of calcium carbonate found in the final PCC product. While the peak at a wavelength of 505 cm<sup>-1</sup> shows the O – Ti – O functional group. Along the wavelength range of 1300 cm<sup>-1</sup> to 700 cm<sup>-1</sup>, it is also possible to observe the absorption of functional groups in the C-H bending framework of the compound.



**Fig. 2:** FTIR results of TiO<sub>2</sub>-PCC and TiO<sub>2</sub>-zeolite nanocomposites

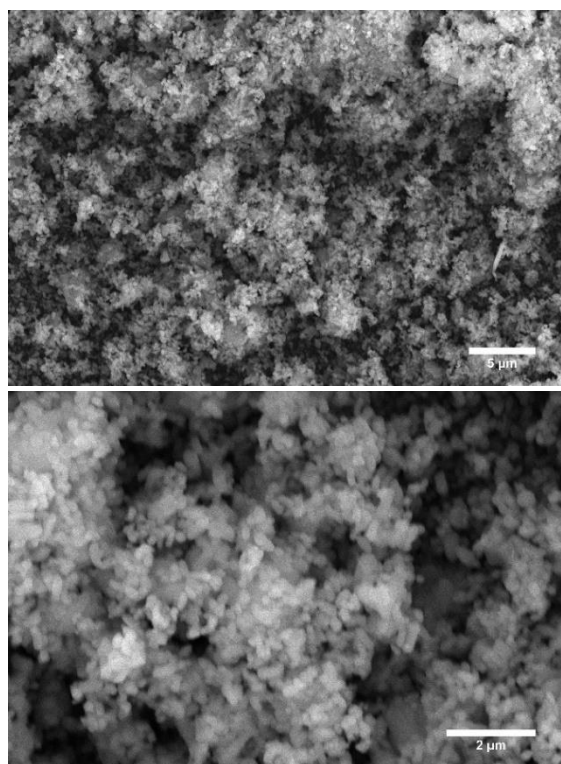
Figure 2 also provides information on the absorption of functional groups on the TiO<sub>2</sub>-Zeolite photocatalyst using FTIR, with peaks observed at wavelengths of 1040 cm<sup>-1</sup>, 692 cm<sup>-1</sup>, and 505 cm<sup>-1</sup>.

The results of characterization with FTIR show that in this curve, there is the absorption of the O – Ti – O functional group in the rutile phase in the peak area at wavelengths of 692 cm<sup>-1</sup> and 505 cm<sup>-1</sup> (Zainal Abidin *et al.*, 2017). Combining TiO<sub>2</sub> with zeolite produces Ti-O-Si bonds at an infrared band of 1040 cm<sup>-1</sup>, and the intensity is proportional to the amount of TiO<sub>2</sub> added to the zeolite structure. The sharp peaks that are

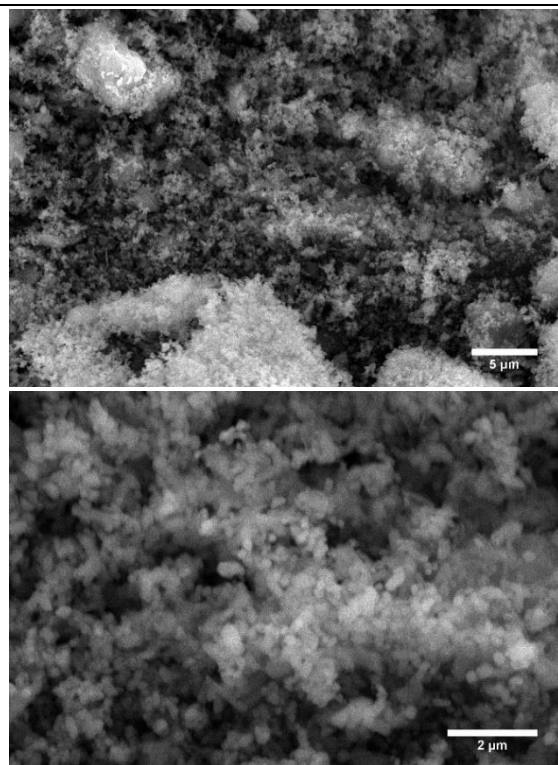
formed can also occur due to the Si–O–Si or Al–O–Al non-symmetric stretching vibrations in the tetrahedral  $\text{SiO}_4$  and  $\text{AlO}_4$  structures that form the zeolite framework (Chong *et al.*, 2015a).

### Scanning Electron Microscope (SEM)

The morphology and nanostructure of the  $\text{TiO}_2$ -PCC nanocomposites (40%  $\text{TiO}_2$ ) were observed in SEM characterization presented in Figure 3.  $\text{TiO}_2$  nanoparticle is observed to cover the surface of PCC. As the PCC is a porous medium, once it is incorporated, the  $\text{TiO}_2$  photocatalyst directly occupies the inside of the pores of the adsorbent. The particle size ranges from 6.11  $\mu\text{m}$  to 7.74  $\mu\text{m}$ . It is also observed that the  $\text{TiO}_2$ -PCC nanocomposite tends to form rock-like shapes with uneven particle sizes. Particle size measurements were performed using image analysis software.



**Fig. 3:** SEM characterization of  $\text{TiO}_2$ -PCC (a) 3000x magnification (b) 7500x magnification



**Fig. 4:** SEM characterization of  $\text{TiO}_2$ -Zeolite (a) 3000x magnification (b) 7500x magnification

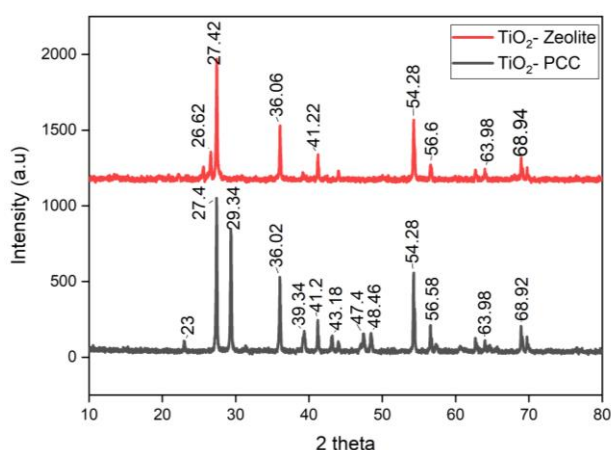
The  $\text{TiO}_2$ -Zeolite SEM image shown in Figure 4 depicts the irregular sphere of  $\text{TiO}_2$  with a slightly larger size than the  $\text{TiO}_2$  nanoparticle found in the  $\text{TiO}_2$ -PCC nanocomposite, with a size range from 8.21  $\mu\text{m}$  to 12.07  $\mu\text{m}$ . It also distributes inside the pores of the zeolite and covers its surface of the zeolite.

### X-Ray Diffraction (XRD)

The analysis by X-ray diffraction of  $\text{TiO}_2$ -PCC is shown in the diffractogram in Figure 5. The peaks that appear at diffraction angles  $2\theta = 27.4^\circ, 36.02^\circ, 41.2^\circ, 54.28^\circ, 56.58^\circ, 63.98^\circ$ , and  $68.92^\circ$  corresponds to Miller indices of (110), (101), (111), (211), (220), (002) and (301) plane, respectively (JCPDS Card No: 21-1276). It refers to rutile  $\text{TiO}_2$  crystal (Copland *et al.*, 2018; Saravy, 2014; Slamet *et al.*, 2020). The peak that appears at  $2\theta = 23.0^\circ, 29.34^\circ, 36.02^\circ, 39.34^\circ, 43.18^\circ, 47.4^\circ$ , and  $48.4^\circ$

indicates the presence of calcite (JCPDS card No. 47-1743). A refers to the content of PCC (calcite crystals) used as an adsorbent/buffer in the TiO<sub>2</sub>-PCC nanocomposite catalyst system (Somarathna *et al.*, 2016). From this diffractogram, it is not certain whether TiO<sub>2</sub> has been dispersed or not in the pores or the external surface of the PCC.

There are several types of crystals in the nanocomposite, namely rutile, calcite, clinoptilolite, and mordenite. Rutile crystals come from TiO<sub>2</sub> compounds, while clinoptilolite and mordenite crystals come from zeolite. The presence of these crystals in the nanocomposite is evident from the XRD results in Figure 5.



**Fig. 5:** XRD characterization of TiO<sub>2</sub>-PCC and TiO<sub>2</sub>-Zeolite

The peaks that appear at diffraction angles  $2\theta = 27.42^\circ, 36.06^\circ, 41.22^\circ, 54.28^\circ, 56.6^\circ, 63.98^\circ$ , and  $68.94^\circ$  corresponds to Miller indices of (110), (101), (111), (211), (220), (002) and (301) plane, respectively (JCPDS Card No: 21-1276). It also refers to rutile TiO<sub>2</sub> crystals (Copland *et al.*, 2018; Saravy, 2014; Slamet *et al.*, 2020). From the XRD analysis, data were obtained that show reflections at  $2\theta = 26.62^\circ, 27.42^\circ$ , and  $36.06^\circ$ , which are characteristic of clinoptilolite and mordenite minerals contained in the zeolite. The diffraction

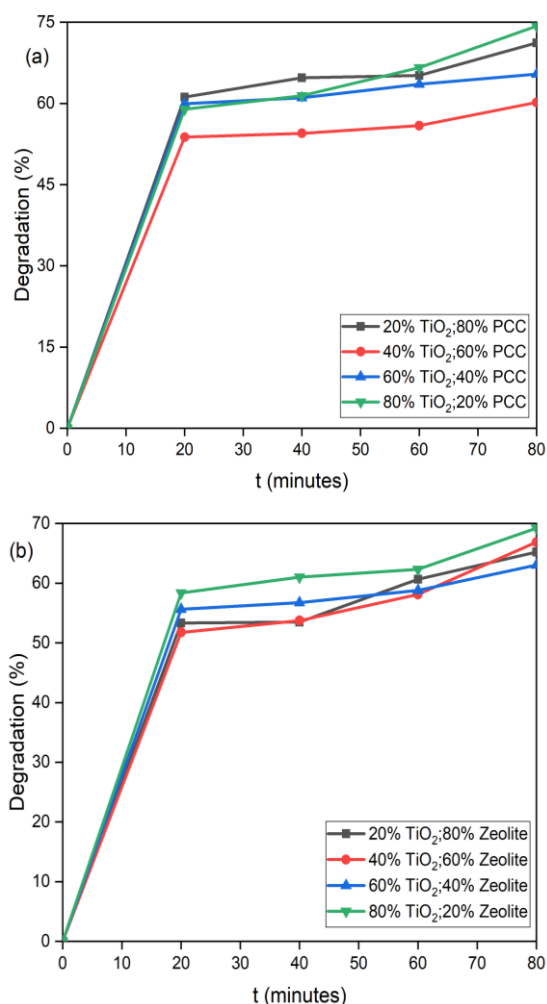
pattern on the zeolite is not very visible due to overlap with TiO<sub>2</sub>. Therefore, the results of the XRD analysis show the dominance of the TiO<sub>2</sub> peak (Fatimah, 2017).

### The Effect of UV Irradiation Time on Phenol Degradation

In this research, experiments on phenol degradation were conducted with various mass ratios to determine the optimum mass ratio in the TiO<sub>2</sub>-PCC nanocomposite. This experiment was performed for 20, 40, 60, and 80 minutes across all concentration variations. Measurements were carried out using a UV-Vis Double-Beam spectrophotometer with an optimum phenol wavelength of 198 nm, using a 4000ppm phenol solution.

Figure 6 illustrates that increasing the duration of UV irradiation leads to a higher percentage of phenol degradation. The TiO<sub>2</sub>-PCC nanocomposite achieved a degradation efficiency of 74.30% after 80 minutes, with an optimal composition ratio of 80% TiO<sub>2</sub> to 20% PCC. A similar trend was observed for the TiO<sub>2</sub>-zeolite nanocomposite, where the highest phenol degradation—69.22%—was also recorded at the composition of 80% TiO<sub>2</sub> and 20% zeolite after 80 minutes of irradiation. According to the research by Slamet *et al.*, testing the performance of nanocomposites degrading phenolic compounds required an irradiation time of 30 minutes for the TiO<sub>2</sub> photocatalyst and 90 minutes for all types of nanocomposites to achieve 100% conversion (Slamet *et al.*, 2020). A 90-minute irradiation time is the optimum time for the TiO<sub>2</sub> nanocomposite photocatalyst process. This condition is consistent with the findings of this study, where the time required to degrade phenol is 80 minutes, and the percentage of phenol degradation appears to increase with

increasing irradiation time. The longer the irradiation time, the more visible light hits the  $\text{TiO}_2$  catalyst, resulting in the production of more  $\text{OH}^\bullet$  radicals to degrade phenol (Alalm *et al.*, 2014).



**Fig. 6:** The effect of UV irradiation time and phenol degradation in  $\text{TiO}_2$ -PCC nanocomposites (a);  $\text{TiO}_2$ -Zeolite nanocomposites (b)

The highest percentages of phenol degradation were observed at the nanocomposite mass ratios of 80%  $\text{TiO}_2$ -20% PCC and 80%  $\text{TiO}_2$ -20% zeolite. This is attributed to the increased amount of catalyst, which enhances the availability of surface-active sites, thereby promoting more efficient photocatalytic activity.

The more active sites on the nanocomposite, the greater the number of photons and phenolic compound molecules that can be adsorbed on its surface (Panuh *et al.*, 2019). Active sites in the nanocomposite are determined by the morphology, size, and distribution of  $\text{TiO}_2$  particles (Tsymbalyuk *et al.*, 2019). In this case,  $\text{TiO}_2$  particles observed in the nanocomposites, as revealed by SEM analysis, tend to exhibit a nanoparticle morphology with sizes below  $1\ \mu\text{m}$ , which may contribute to the enhanced photodegradation performance.

From an application perspective, the use of  $\text{TiO}_2$ -PCC nanocomposites offers greater value in terms of catalyst separation.  $\text{TiO}_2$  in the form of a  $\text{TiO}_2$  - PCC nanocomposite tends to form a precipitate at the end of the reaction, making it easier to separate from the liquid waste system (Slamet *et al.*, 2020).

Compared to pristine zeolite materials, both  $\text{TiO}_2$ -PCC and  $\text{TiO}_2$ -zeolite nanocomposites exhibit significantly higher performance in phenol removal, achieving removal capacities in the range of 93–100 mg/g within 80 minutes. In contrast, zeolitic tuff has been reported to remove only 34.5 mg/g at  $25^\circ\text{C}$  in 90 minutes through adsorption mechanism (Yousef *et al.*, 2011), ZSM-5 zeolite achieved 24.41 mg/g in 120 minutes (Liu & Lu, 2020), and zeolitic volcanic tuff exhibited much lower removal capacities, ranging from 0.12 to 0.53 mg/g at  $25^\circ\text{C}$  (Bizerea Spiridon *et al.*, 2013). Meanwhile, no phenol adsorption capacity has been reported for pristine PCC, suggesting that its enhanced performance is primarily due to its synergistic interaction with  $\text{TiO}_2$  in the nanocomposite structure.

While this study demonstrates the effectiveness of  $\text{TiO}_2$ -PCC and  $\text{TiO}_2$ -zeolite nanocomposites in phenol degradation under UV irradiation, it does not include



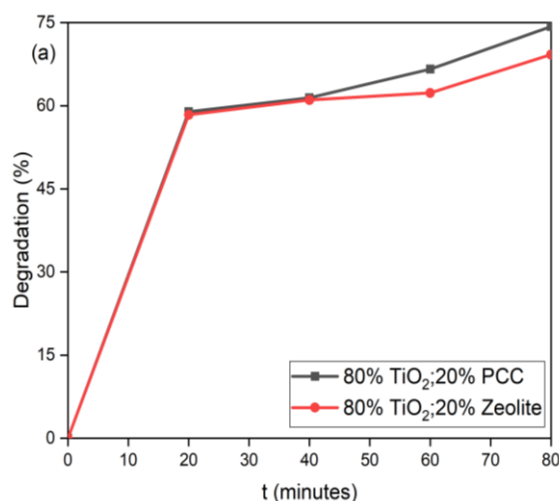
comparative adsorption tests conducted in the absence of light (dark conditions). As both PCC and zeolite are known adsorbent materials, part of the observed phenol removal may be attributed to adsorption rather than photocatalysis alone. The rapid decrease in phenol concentration during the first 30 minutes is likely dominated by adsorption onto the surface of the nanocomposites. This initial phase is commonly observed in systems combining porous supports and photocatalysts—where the porous adsorbent (e.g. zeolite or PCC) quickly concentrates target molecules before photocatalytic degradation takes over. A similar observation has been reported in a study on TiO<sub>2</sub>-based composites, where adsorption-derived removal is significant in the early stages (Bahrudin, 2022).

### Nanocomposite Adsorbents Performance in Phenol Degradation

The use of different adsorbents in the synthesis of TiO<sub>2</sub> nanocomposites also results in different degradation of phenol. Based on Figure 7, it can be observed that the TiO<sub>2</sub>-PCC nanocomposite degrades phenol more effectively than the TiO<sub>2</sub>-Zeolite nanocomposite. A significant difference in degradation ability occurred after 40 minutes. Both nanocomposites showed an increase in phenol degradation over time, but TiO<sub>2</sub>-PCC is capable of degrading more phenol than TiO<sub>2</sub>-Zeolite in the same period of up to 80 minutes.

This is based on the research conducted by Jati *et al.* who found that the percent degradation of the TiO<sub>2</sub>-PCC nanocomposite was greater than that of the TiO<sub>2</sub>-Zeolite nanocomposite in the degradation of chromium and organic matter (Jati *et al.*, 2012). This is also due to the results of SEM characterization, which show that PCC has a

smaller particle/crystal size compared to zeolite, so that it can disperse TiO<sub>2</sub> nanocomposites better (evenly) (Panuh *et al.*, 2019).



**Fig 7.** Comparison of the degradation ability of TiO<sub>2</sub>-PCC and TiO<sub>2</sub>-Zeolite

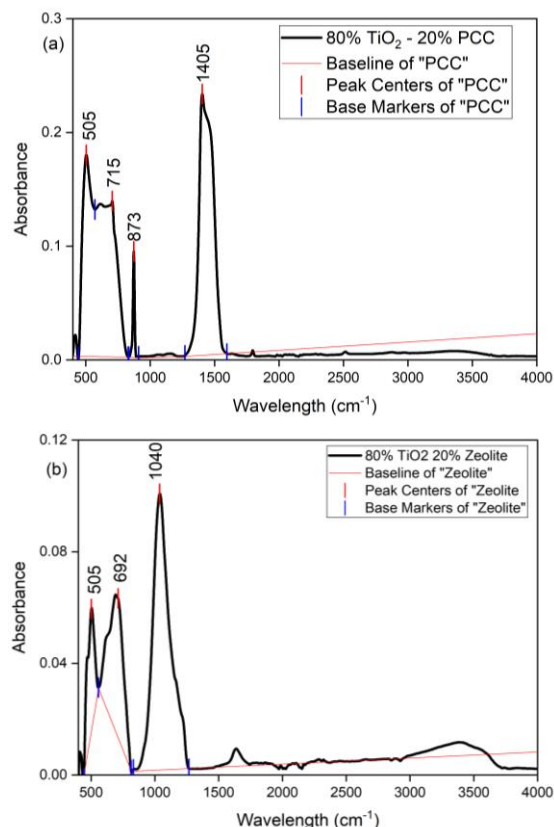
### The Presence of a Functional Group Area in Phenol Degradation

The functional group area also affects the nanocomposite's ability to degrade phenol. This is evident in Figure 8 and Table 2, where the TiO<sub>2</sub>-PCC nanocomposite exhibits a larger functional group area than TiO<sub>2</sub>-Zeolite. The wider the functional group area, the greater the nanocomposite's ability to adsorb phenol. The more phenol that is adsorbed, the more phenol is degraded.

In TiO<sub>2</sub>-Zeolite, the functional group can be identified by the presence of peaks at wavelengths of 1040 cm<sup>-1</sup>, 692 cm<sup>-1</sup>, and 505 cm<sup>-1</sup>. The TiO<sub>2</sub>-PCC nanocomposite contains functional groups of O-Ti-O in the rutile phase, O-H from a water molecule, or Ti-OH, and there is a bending vibration of the carbonate ion of calcite. The functional groups contained in TiO<sub>2</sub>-zeolite are O – Ti – O and Ti-O-Si bonds. The sharp peaks that are formed can also occur due to the Si-O-Si or Al-O-Al non-symmetric stretching



vibrations in the tetrahedral  $\text{SiO}_4$  and  $\text{AlO}_4$  structures that form the zeolite framework.



**Fig. 8:** Functional group area on nanocomposite  $\text{TiO}_2$ -PCC (a)  $\text{TiO}_2$ -Zeolite (b)

**Table 2.** Functional group area on  $\text{TiO}_2$ -PCC nanocomposite

The peak at Wavelength ( $\text{cm}^{-1}$ )	Functional Group	Area
<b><math>\text{TiO}_2</math>-PCC</b>		
505	O-Ti-O	16.332
715	$-\text{CO}_3$	24.870
873	$-\text{CO}_3$	1.520
1405	$-\text{OH}$ / Ti-OH	29.902
<b>Total Area</b>		<b>72.624</b>
<b><math>\text{TiO}_2</math>-Zeolite</b>		
505	O-Ti-O	2.960
692	O-Ti-O	6.900
1040	Ti-O-Si	15.876
-	-	-
<b>Total Area</b>		<b>25.736</b>

The area of the functional group may affect the adsorption mechanisms that occur on the nanocomposite surface. Some research has found that the adsorption of dyes and other pollutants on  $\text{TiO}_2$ -zeolite composites can be significantly affected by the presence of specific functional groups that enhance the interaction with the adsorbates (Chong *et al.*, 2015b; Kusumawardani *et al.*, 2025; Motamedi *et al.*, 2022). The functional groups can also affect the hydrophobic/hydrophilic properties of the composites, which in turn influence the adsorption behavior (Rani & Shanker, 2023). In this work, the functional groups likely facilitate the adsorption of phenol to the surface of nanocomposites, which further enables the active sites and surface area of  $\text{TiO}_2$  nanoparticles to perform photodegradation through the photocatalysis process.

## CONCLUSIONS

The nanocomposites  $\text{TiO}_2$ -PCC and  $\text{TiO}_2$ -Zeolite have been successfully synthesized through a wet chemical process. The FTIR, SEM, and XRD data verify the properties of the nanocomposites, showing that the impregnation of  $\text{TiO}_2$  into zeolite and PCC was accomplished effectively. The application of the nanocomposite for phenol degradation shows that the highest percentage of phenol degradation was achieved with a mass ratio of 80%  $\text{TiO}_2$ -20% PCC, and 80%  $\text{TiO}_2$ -20% zeolite. This is due to the increasing number of catalysts, which in turn increases the number of active sites for capturing photons and phenol molecules.  $\text{TiO}_2$ -PCC nanocomposite can degrade more phenol than  $\text{TiO}_2$ -Zeolite nanocomposite, with the degradation of 74.30%. The  $\text{TiO}_2$ -zeolite nanocomposite can degrade phenol

by only 69.22%. This is due to PCC having a smaller particle/crystal size compared to zeolite, so that it can disperse the TiO<sub>2</sub> nanocomposite better (evenly). The larger TiO<sub>2</sub>-PCC functional group area also contributes to the absorption of phenol, so the possibility is that the amount degraded increases.

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