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THE REMOVAL OF COD, TDS, TSS, AND COLOR FROM BATIK WASTEWATER USING OZONATION-ADSORPTION IN A PACKED-BED SYSTEM WITH HCL ACTIVATED ZEOLITE

S. Susilowati[1,2,✉], M. A. Nafii'u Hafid[1], H. Z. Syadana[1], K. Sumada[1], A.P.Bayuseno[3], and W. W. Schmahl[4]

[1]Department of Chemical Engineering, University Pembangunan Nasional "Veteran" Jawa Timur, Surabaya-60294 (East Java), Indonesia
[2]Postgraduate Program of Environmental Science, University Pembangunan Nasional "Veteran" Jawa Timur, Surabaya-60294 (East Java), Indonesia
[3]Department of Mechanical Engineering, Diponegoro University, Semarang-50275 (Central Java), Indonesia
[4]Department of Earth and Environmental Sciences, Ludwig Maximilians University of Munich, Munich-80539 (Bavaria), Germany
✉Corresponding author: susilowati.tk@upnjatim.ac.id

ABSTRACT

The batik industry generates highly polluted wastewater containing recalcitrant organic compounds and intense color, which are inadequately treated by conventional methods. This study addresses this limitation by developing a hybrid ozonation–adsorption process using HCl-activated natural zeolite in a packed-bed system to enhance pollutant removal efficiency. The integration of advanced oxidation and adsorption is designed to overcome mass-transfer limitations and incomplete degradation commonly observed in single-treatment processes. Under optimal conditions (packing height of 25 cm and contact time of 40 min), the system achieved removal efficiencies of 93.27% for COD, 93.39% for TDS, 99.98% for TSS, and 98% for color. Acid activation increased the zeolite surface area, improving adsorption capacity, while ozonation generated hydroxyl radicals that enhanced the breakdown of complex organics. FTIR analysis confirmed structural and functional group changes, including shifts in hydroxyl (–OH), carbonyl (C=O), and Si–O–Si/Si–O–Al bands, indicating interactions between the zeolite surface and organic pollutants during the treatment process. The adsorption process followed the Langmuir isotherm model (R^2 = 0.9998), indicating monolayer adsorption on a homogeneous surface. This study contributes to wastewater treatment by demonstrating a synergistic mechanism between oxidation and adsorption that enhances treatment performance compared to conventional approaches. The findings provide a scalable, efficient solution for dye-containing industrial wastewater and lay a foundation for future studies on process optimization and reactor scale-up.

Keywords: Adsorption, Batik Wastewater, COD, Color Removal, Hybrid Ozonation, Packed Bed Reactor.

INTRODUCTION

Batik represents an important cultural identity of Indonesia; however, its manufacturing processes generate complex wastewater containing dyes and chemical additives.[1,2] A general batik production facility generates substantial liquid wastewater from dyeing and bleaching activities, which contains synthetic dyes and persistent organic compounds.[3,4] The COD, TDS, TSS, and color parameters often exceed established environmental quality standards.[5,6] Regulatory standards for industrial wastewater generally impose strict limits on parameters such as organic load, suspended solids, dissolved solids, and color. In many cases, these thresholds are exceeded, indicating the need for improved, more efficient treatment technologies.[7] Conventional approaches, such as coagulation–flocculation, filtration, and biological treatment, have been widely applied.[8] These methods remain limited in removing recalcitrant organic compounds and synthetic dyes due to their complex molecular structures and low biodegradability.[9] Previous studies have also reported that single-treatment systems often suffer from

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mass-transfer limitations and incomplete degradation, resulting in suboptimal treatment performance. Therefore, there is a clear research gap in developing an integrated treatment system that can simultaneously enhance oxidation and adsorption processes to achieve higher removal efficiency.[10,11] Ozonation offers a promising alternative because ozone (O_3) is a strong oxidant capable of decomposing pollutants and generating highly reactive hydroxyl radicals (•OH).[12] Its effectiveness can be enhanced by integrating adsorption with HCl-activated zeolite in a packed-column configuration, in which the zeolite adsorbs residual contaminants, while glass-marble packing increases the contact area and prolongs the interaction between ozone and the adsorbent.[13] Glass marbles are selected for their uniform gas–liquid distribution, cost efficiency, and durability under long-term operation.[14] Activation of natural zeolite with 2 M HCl removes pore-blocking impurity ions and increases active surface area through soaking, washing with deionized water, and controlled drying.[15] Moreover, ozonation reduces sludge formation because it requires minimal chemical addition and simultaneously provides a disinfection effect, decreasing microbial content and improving effluent safety and cleanliness.[16] Ozone undergoes several steps to form powerful cleaning particles in water. First, ozone reacts with water to make special ions:

$$O_3 + H_2O \rightarrow HO_3^+ + OH^-$$

Next, HO_3^+ joins with OH^- to make two hydroperoxyl (HO_2) particles:

$$HO_3^+ + OH^- \rightarrow 2HO_2$$

Then, ozone reacts with HO_2 to create a hydroxyl particle (HO*) and two oxygen molecules:

$$O_3 + HO_2 \rightarrow HO^* + 2O_2$$

The very active HO* particles further react with HO_2 to form water and oxygen:

$$HO^* + HO_2 \rightarrow H_2O + O_2$$

Under alkaline conditions, degradation of complex compounds is more effective. Higher OH^- concentration increases radical formation and acts as a strong oxidizing agent. Abundant OH^- results in greater HO* production, improving the oxidation of organic matter in batik wastewater.[17] Calcium (Ca^{2+}) and magnesium (Mg^{2+}) levels drop due to ion exchange between the zeolite's cations and hydrogen ions (H^+) released during HCl activation.[18] During adsorption, wastewater with Ca^{2+} and Mg^{2+} passes through the zeolite column. Ion exchange occurs at this stage:

$$H_2Z + Ca^{2+} \rightarrow CaZ + 2H^+$$
$$H_2Z + Mg^{2+} \rightarrow MgZ + 2H^+$$

Regenerate the zeolite using HCl after it becomes saturated, thus restoring its acidic form via:

$$CaZ + 2HCl \rightarrow H_2Z + CaCl_2$$
$$MgZ + 2HCl \rightarrow H_2Z + MgCl_2$$

In this mechanism, Ca^{2+} and Mg^{2+} are released as soluble chlorides. H^+ returns to the zeolite framework, so the material can be reused in later adsorption cycles.[19] This study addresses the need for a more effective treatment approach for batik wastewater by investigating a hybrid ozonation–adsorption system using HCl-activated natural zeolite in a packed-bed configuration. The specific objective is to evaluate how packing height and contact time influence the removal of COD, TDS, TSS, and colour, particularly under conditions where conventional treatment methods are less effective in removing recalcitrant organic compounds and synthetic dyes. By combining oxidative degradation with adsorption enhancement, this approach is expected to improve treatment efficiency and offer a practical basis for developing more reliable wastewater treatment strategies for dye-containing industrial effluents. Combining ozonation and adsorption is expected to improve the removal of COD, TDS, TSS, and color compared to either method alone.[20] This study examines how packing media and activated zeolite affect pollutant reduction in the ozonation system, thereby supporting the development of efficient and reliable wastewater treatment technology.

EXPERIMENTAL

Material and Methods

The materials used in this study consisted of batik wastewater as the primary sample, natural zeolite activated with 2 M HCl as the adsorbent, and glass marbles as the packing media in the packed column

system. Distilled water was used for washing and dilution. The mass of activated zeolite applied in the reactor was fixed at 400 g, and 750 mL of wastewater was used per batch, with a 1:2 dilution ratio (wastewater to distilled water).

General Procedure

Activation of Natural Zeolite with 2M HCl Solution

The zeolite activation process began by grinding the natural zeolite until it passed through a 60-mesh sieve, followed by washing and thermal treatment with distilled water to remove surface impurities. The zeolite is then dried at 110°C for 3 hours before being mixed with a 2M HCl solution at a ratio of 3:1 (v/m) (volume of 2M HCl solution to mass of zeolite). The mixture is stirred with a magnetic stirrer at 250 rpm for 2 hours at approximately 30°C. Subsequently, the filtrate is discarded, and the zeolite residue is washed repeatedly until the pH is neutral. The activated zeolite is dried again at 200°C for 2 hours. Zeolite activated with 2M HCl is then used as an adsorbent to reduce pollutant concentrations in wastewater.

Implementation of a Series of Tools

The experiments were performed using two reactor configurations: a column packed with activated zeolite without packing material, serving as a comparative control, and a column packed with activated zeolite with glass beads. The experimental variables comprised the height of the glass-bead packing layer (0, 5, 10, 15, 20, and 25 cm) and the ozone contact time (20, 25, 30, 35, and 40 min). All operating parameters were held constant, including an activated zeolite mass of 400 g, an ozone dosage of 400 mg/h, and an operating temperature of approximately 30°C.

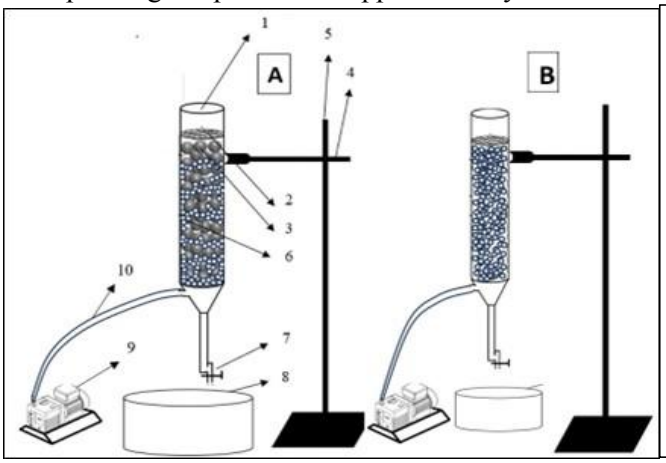

Figure description:

1. Packed-bed column
   a) HCl-activated zeolite (2 M) with packing material
   b) HCl-activated zeolite (2 M) without packing material
2. Packed filter (sieve)
3. Packing media (glass marbles)
4. Clamp
5. Stand
6. Activated zeolite
7. Valve
8. Final storage tank
9. Ozone generator

Fig.-1: Series of Ozonation and Waste Adsorption Packed Column Devices with and without Packing Material

Wastewater samples corresponding to the designated variables are introduced into the column reactor according to the experimental configuration. [21] Under conditions in which activated zeolite with glass-bead packing is employed, the packing material is added to the reactor according to the specified height variation. Ozone is then supplied from the generator for the predetermined contact time. The same procedure is applied to the configuration utilising activated zeolite without glass-bead packing.

Detection Method

The characteristics of zeolite before and after activation were analyzed using the Brunauer–Emmett–Teller (BET) method to determine changes in surface area and pore structure.[22,23] After being used in wastewater treatment, the zeolite sample, both with and without glass-bead packing, was further analyzed using SEM-EDX and FTIR to investigate surface morphology, elemental composition, and functional groups, including changes caused by HCl activation and interaction with wastewater.[24] Furthermore, the quality of batik wastewater before and after treatment was assessed using COD, TDS, TSS, and color (PtCo) parameters. COD was determined by potassium dichromate oxidation under acidic, heated conditions, with oxygen demand calculated from titration or absorbance values as an indicator of organic compound degradation.[25] TDS was measured using a conductivity meter and expressed in $mg.L^{-1}$.[26] TSS

was analyzed gravimetrically by filtration, drying, and weighing until a constant mass was obtained, while color was measured using a UV–Vis spectrophotometer calibrated with the Pt–Co standard at wavelengths corresponding to the dye characteristics in the wastewater.

RESULTS AND DISCUSSION

Initial Characteristics of Batik Wastewater

[27] Before treatment, the wastewater was analyzed to determine its initial characteristics, including COD, TDS, TSS, and color. As presented in Table-1, the measured values were 5880 $mg.L^{-1}$ for COD, 10288 $mg.L^{-1}$ for TDS, 11980 $mg.L^{-1}$ for TSS, and 1501 PtCo units for color.

Table-1: Preliminary Analysis Results of Batik Wastewater Before Treatment

Parameter	Analysis Result ($mg.L^{-1}$)
COD (Chemical Oxygen Demand)	5880
TDS (Total Dissolved Solids)	10288
TSS (Total Suspended Solids)	11980
Color (PtCo Units)	1501

These values are well above the permissible discharge limits, indicating a substantial pollution load in the batik wastewater. The elevated COD value reflects a high concentration of oxidizable organic compounds, mainly from synthetic dyes and auxiliary chemicals used in batik production. The high TDS indicates the presence of dissolved substances such as salts, residual reagents, and dye-processing by-products, while the TSS shows a large amount of suspended particles that may increase turbidity and sedimentation if discharged untreated. The color intensity of 1501 PtCo units further confirms the dominance of dye compounds, which are often among the most difficult pollutants to remove. Overall, these results highlight the need for a more effective treatment method. The combined ozonation–adsorption process studied here is expected to remove both dissolved and suspended pollutants while improving color reduction. These findings also relate to Sustainable Development Goals (SDG-6) by emphasising the need for better wastewater management and cleaner water resources in batik production.

Characterisation of Natural Zeolite Surface Area Using BET

Changes in surface characteristics, particularly surface area and pore distribution, of natural zeolite were evaluated in its initial condition and after activation with 2M HCl.

Table-2: Surface Area of Natural Zeolite Before and After Activation

Zeolite Type	Surface Area (m^2/g)
Natural zeolite	213.470
Natural zeolite activated with 2M HCl	237.722

BET analysis results (Table-2) indicate that the surface area of natural zeolite increased after activation, rising from 213.470 m^2/g to 237.722 m^2/g. This increase of approximately 24.252 m^2/g indicates a clear improvement in the material's physicochemical properties. The higher surface area suggests that the activation process effectively opened previously blocked pores, thereby improving pore accessibility. In untreated zeolite, impurities and exchangeable cations tend to occupy pore spaces, limiting adsorption performance. After activation, these obstructions are reduced, making the surface more accessible for adsorption. From a qualitative perspective, the activated zeolite exhibits a more developed pore structure, whereas quantitatively, the increase in surface area reflects a greater number of active sites. This condition is beneficial for adsorption processes, as a larger surface area generally enhances pollutant removal capacity. HCl also plays an important role in this process. Hydrogen ions (H^+) generated during activation replace native cations such as Na^+, Ca^{2+}, and Mg^{2+} within the zeolite framework through ion exchange. This not only removes pore-blocking species but also improves the reactivity of the zeolite surface.

Surface Morphology of Zeolite after Combined Processes

Scanning electron microscopy (SEM) was employed to observe the surface morphology of the activated zeolite after its application in batik wastewater treatment, both in the presence and absence of packing material (Fig-3).

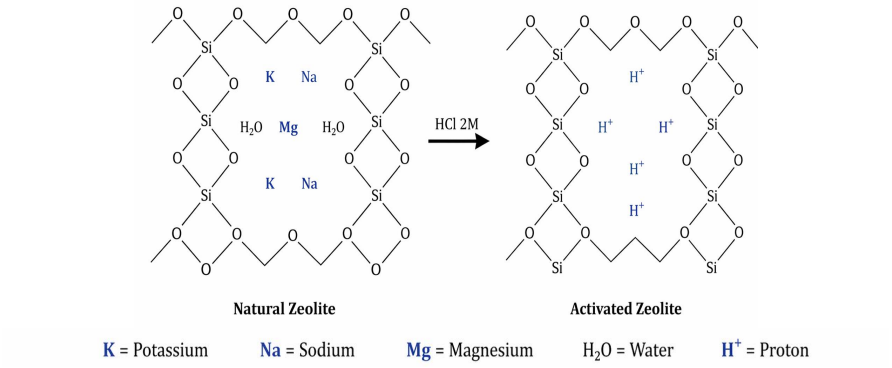

Fig.-2: Reaction Mechanism of Natural Zeolite Activated with 2M HCl

As illustrated in Fig-3(a), the zeolite in the absence of packing displays an irregular and coarse surface, accompanied by noticeable gaps between particles. Conversely, Fig-3(b) demonstrates that packing results in a more coated surface, with a relatively uniform layer of deposited substances covering the particle matrix. This suggests that the packing material increased contact time and interaction among ozone, wastewater, and zeolite, thereby promoting the adsorption of dissolved organic compounds, suspended particles, and oxidation products onto the zeolite surface. This improvement in surface interactions and pollutant retention demonstrates the system's potential to enhance effluent quality and reduce contaminant discharge, which is directly relevant to efforts to improve water quality and control industrial wastewater pollution.

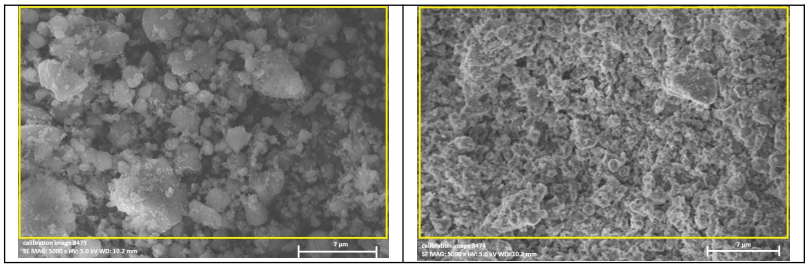

Fig.-3: SEM Micrographs of Zeolite Surface After Batik Wastewater Treatment: (a) Surface Zeolite without Material Packing, (b) Surface Zeolite with Material Packing

Elemental Analysis

In Figure-4, the EDX spectrum of the unpackaged zeolite is dominated by oxygen and silicon, with contents of 74.90 wt% and 15.37 wt%, respectively, which reflects the typical aluminosilicate framework of natural zeolite. After treatment in the packed system, the oxygen content decreased to 67.34 wt%, whereas silicon increased to 20.90 wt%, indicating surface modification and greater exposure of silicate sites after the combined ozonation–adsorption process. In addition, the increase in carbon content from 3.36 wt% to 3.66 wt% suggests the presence of adsorbed organic species from batik wastewater on the zeolite surface. The increase in aluminium content to 4.33 wt% also supports changes in surface composition after treatment. Overall, these EDX results are consistent with the SEM observations and indicate a stronger interaction between the zeolite surface and wastewater constituents, thereby improving effluent quality and reducing pollutant discharge. This outcome is relevant to broader efforts to improve water quality by reducing industrial wastewater contamination, which aligns with sustainable wastewater management objectives.

Characterisation of Zeolite Surface Functional Groups

Figure-5 presents FTIR analysis showing variations in band intensity and shifts in absorption bands associated with functional groups in zeolite after contact with batik waste, both in systems with and without fillers. The absorption band in the range of 3400–3600 cm^{-1}, attributed to the stretching vibration

of hydroxyl (-OH) groups, decreased in intensity in the zeolite with packing, indicating interactions between hydroxyl groups and polar compounds in the wastewater via hydrogen bonding. The band observed around 1650 cm^{-1}, associated with H–O–H bending and carbonyl (C=O) vibrations, also showed reduced intensity, suggesting enhanced adsorption of water molecules and organic compounds resulting from dye degradation.

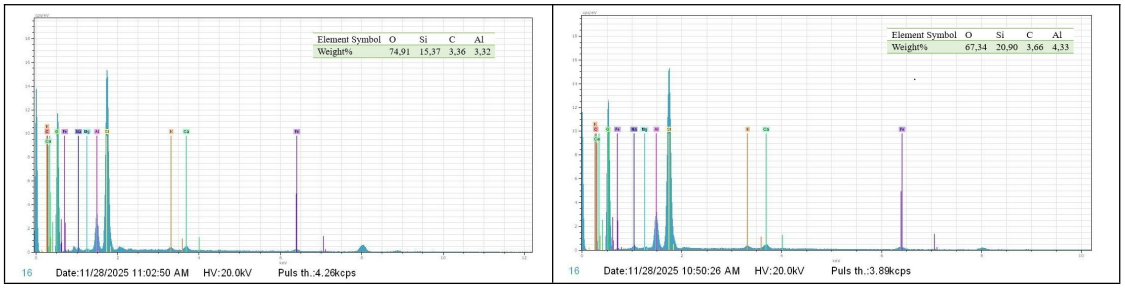

Fig.-4: EDX Analysis Showing The Elemental Composition of Zeolite After Adsorption of Batik Wastewater: (a) Surface Zeolite without Material Packing, (b) Surface Zeolite with Material Packing

In addition, the absorption band around 1396 cm^{-1}, attributed to methyl ($–CH_3$) functional groups, showed a reduced transmittance in the packed system, suggesting a higher uptake of nonpolar organic substances. Meanwhile, the typical zeolite framework band in the 1000–1100 cm^{-1} range, corresponding to Si–O–Si and Si–O–Al vibrational modes, showed a slight shift, suggesting possible interactions between the zeolite surface and metal ions or organic components in the wastewater. Overall, these results demonstrate that incorporating a packing system enhances adsorption efficiency by increasing the effective surface area of contact and improving mass-transfer distribution. This FTIR evidence decisively strengthens the mechanistic understanding of the combined process and clearly demonstrates that the improved removal performance is reflected not only in macroscopic water-quality data but also in molecular-level surface interactions, confirming its substantial practical relevance for achieving cleaner wastewater discharge.

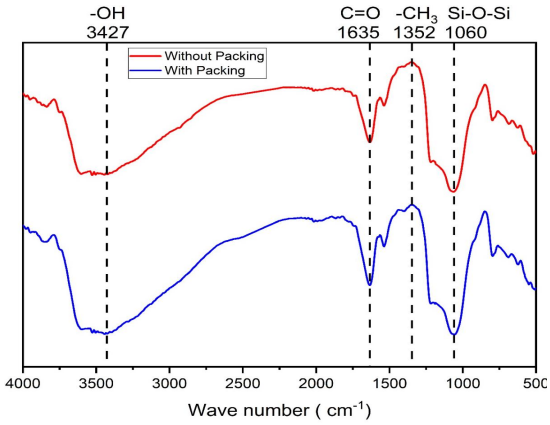

Fig.-5: FTIR Spectra of Zeolite Surface with and without the use of Packing Material

Decrease in TDS, TSS, Color (PtCo) and COD Levels in Filtrate Result

As indicated in Table-3, significant reductions in TDS, TSS, and color were achieved within 40 minutes at a bed height of 25 cm, with removal efficiencies of 93.39% (680 $mg.L^{-1}$) for TDS, 99.98% (2 $mg.L^{-1}$) for TSS, and 98% (30 $mg.L^{-1}$) for color. The incorporation of packing material and zeolite contributes to a lower wastewater flow velocity, thereby extending residence time and enhancing contact among the wastewater, zeolite surface, and ozone bubbles. Moreover, a more compact bed structure minimizes channeling effects, promoting a more uniform gas–liquid–solid interaction and improving ozone mass transfer efficiency. In addition, the packing also functions as a mechanical filtration medium that helps capture suspended particles (TSS) and colour-carrying colloidal complexes, making them easier to absorb.[28] Dissolved ozone oxidises complex organic compounds and breaks the chromophore bonds in

dyes, producing simpler and more polar fragments that are easier for the zeolite to absorb. Ozone activation by the zeolite surface also forms highly reactive hydroxyl radicals (•OH), accelerating the degradation of dissolved and particulate pollutants while simultaneously increasing the reduction of TDS, TSS, and color.

[29] Based on the decrease in color analysis parameters (ptco units), optimal results were obtained at 40 minutes at each packing height, followed by a COD test. Based on the COD test results at an ozone contact time of 40 minutes, COD reduction increased with increasing packing height, indicating the important role of packing in enhancing the effectiveness of the ozonation-adsorption process. The system without filler produced the lowest efficiency of 89.6% with a residual COD of 612.2 $mg.L^{-1}$, due to more limited gas-liquid contact and less than optimal interaction between ozone, waste, and zeolite. The addition of filler increased the efficiency to 93.4% at a height of 25 cm with a minimum COD of 395.4 $mg.L^{-1}$, due to increased hydraulic resistance and residence time in the column. The filler triggered ozone bubble fragmentation, thereby expanding the gas-liquid interface area and increasing the solubility of ozone in the liquid phase. Simultaneously, the filler acts as a filtration medium and provides additional area for adsorption, while ozone oxidizes complex organic compounds into smaller, more polar intermediates that are more easily absorbed by active zeolite. The combination of increased ozone mass transfer, longer contact time, and more effective adsorption explains why the layered system produces higher COD reduction compared to the column without filler.[30]

Table-3: Decrease in TDS, TSS, Colour (Unit PtCo), and COD Levels in Filtrate Result

Packing (cm)	Contact Time (minutes)	Decrease Results			
		TDS ($mg.L^{-1}$)	TSS ($mg.L^{-1}$)	Color (Unit PtCo)	COD ($mg.L^{-1}$)
Without Packing Material	20	1072	48	102	
	25	1045	36	77	
	30	1010	32	60	
	35	999	30	50	
	40	960	28	45	612.2
5	20	945	32	72	
	25	938	25	58	
	30	915	21	50	
	35	909	19	44	
	40	902	18	40	596.9
10	20	888	16	53	
	25	868	14	43	
	30	861	13	41	
	35	832	11	38	
	40	820	9	36	512
15	20	802	12	34	
	25	785	9	33	
	30	772	7	32	
	35	764	5	31	
	40	755	4	30	489.2
20	20	740	9	40	
	25	730	7	36	
	30	723	5	34	
	35	716	3	33	
	40	710	3	32	436.2
25	20	700	3	36	
	25	695	3	33	
	30	685	2	32	
	35	683	2	31	
	40	680	2	30	395.4

This demonstrates that the combined ozonation–adsorption system effectively reduces pollutant concentrations, thereby enhancing effluent quality and supporting more sustainable wastewater treatment

practices. These improvements are directly aligned with Sustainable Development Goals (SDG) 6, particularly in achieving better water quality by reducing pollution from industrial wastewater and promoting effective treatment technologies.

Isothermal Adsorption

An adsorption isotherm analysis was conducted to evaluate the interaction between HCl-activated zeolite and pollutant species in the liquid phase. In this work, the Langmuir isotherm was used to interpret the adsorption mechanism. It characterises monolayer coverage on a homogeneous surface, which aligns with the structural properties of acid-activated zeolite. This zeolite has enhanced pore systems and active sites. The model assumes that each site binds a single adsorbate molecule, with no interaction between neighbouring adsorbed species. Using the dataset in Table-4, we used the adsorbent mass (m) and the quantity of dye adsorbed (x) to derive parameters such as C, log C, C/(x/m), and log (x/m). These parameters were used to create the Langmuir isotherm by plotting C versus C/(x/m). The resulting linear relationship between C/Cm and the equilibrium concentration (C) is shown in Fig-6 and described by the equation y = 0.0004x - 0.0009. The correlation coefficient (R^2) is 0.9998, indicating excellent agreement with the model. This strong linearity supports the conclusion that adsorption occurs as a monolayer on a surface with uniform active sites. From the slope (1/a), the maximum adsorption capacity (a) was estimated at 2500 mg. g^{-1}. The intercept, corresponding to 1/(a.b), yielded a Langmuir constant (b) of −0.4444. These results suggest that the activated zeolite possesses a strong adsorption affinity and high capacity for ozonated organic compounds. Furthermore, these findings support broader initiatives to improve water quality by effectively removing persistent industrial pollutants. In this regard, the proposed ozonation–adsorption system contributes to sustainable water management by offering a more efficient and reliable treatment approach.

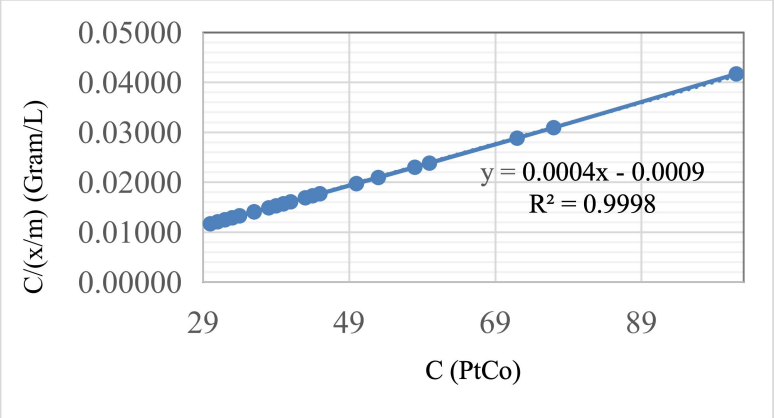

Fig.-6: Langmuir Adsorption Isotherm Expressed as a Relationship between C(PtCo) and C/(x/m)

CONCLUSION

This study showed that the packed-column ozonation–adsorption system using HCl-activated zeolite effectively removed COD, TDS, TSS, and color from batik wastewater. Significant pollutant reduction was observed when the system was operated at a packing height of 25 cm and a retention time of 40 minutes. Under these conditions, removal efficiencies of 93.27% (COD), 93.39% (TDS), 99.98% (TSS), and 98.00% (color) were obtained, indicating that the process operates efficiently at its optimal configuration. The adsorption behavior followed the Langmuir model (R^2= 0.9998), indicating monolayer adsorption. These findings demonstrate that the combined process can improve effluent quality and reduce pollutant discharge, providing a practical basis for more reliable industrial wastewater treatment. Such performance is particularly important for supporting cleaner water management and reducing the environmental burden associated with industrial effluents.

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CONFLICT OF INTERESTS

The authors affirm that this work was conducted without any conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

S Susilowati https://orcid.org/0000-0002-2287-429X
Moch. Afrizal Nafii'u Hafid https://orcid.org/0009-0005-3774-8885
Haqi Zain Syadana https://orcid.org/0009-0009-9996-6588
Ketut Sumada https://orcid.org/0009-0002-0190-3968
Athanasius P Bayuseno https://orcid.org/0000-0002-0882-4480
Wolfgang W. Schmahl https://orcid.org/0000-0002-0165-5952

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