

Article

Decolourisation of Real Industrial and Synthetic Textile Dye Wastewater Using Activated Dolomite

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Abstract: Textile effluent accounts for 22% of the total industrial wastewater produced in Malaysia. Due to their ubiquitous use in organic dyes, inefficiently treated textile wastewaters pose environmental and health hazards. Colour, chemical oxygen demand, biochemical oxygen demand, toxic metals and microbes are the commonly targeted water quality parameters in untreated textile fluids. Furthermore, their non-biodegradability and high colour intensity may reduce aquatic diversity by blocking the sunlight. Recently, physical treatment, principally adsorption, has been conducted. Dolomite has additional features, such as performing as a heavy metal and microbe remover. This study employed dolomite for treating textile dye wastewater from a commercial textile manufacturer and synthetic effluent containing methyl orange. Different sizes of dolomite were activated at different temperatures and subsequently added to the water samples in varying amounts. After 2 h of agitation at 100 rpm and sedimentation for 24 h, their absorbance reading was taken. Their morphological, decolourisation percentage, chemical oxygen demand reduction percentage and bacterial growth post-treatment were examined. Despite variances in dolomite's capacity to decolourise colours, the treatment proved effective in decolourising dyes, removing chemical oxygen demand and reducing bacterial growth. The most significant percentages of decolourisation observed were 98.7% for real textile dye wastewater (RTDW) and 78.0% for synthetic textile dye wastewater (STDW), while for chemical oxygen demand, reductions were 66.7% for RTDW and 73.9% for STDW, respectively. As for microbe growth inhibition, the highest growth reduction percentages were 99.7% and 98.6% for RTDW and STDW, respectively.

Keywords: activated dolomite; decolourisation; textile dye wastewater; adsorption; wastewater treatment



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1. Introduction

The textile industries are developing rapidly in Malaysia and have significantly contributed to economic growth in the past ten years. Dyeing is one of the crucial steps in the process of making garments and clothes, using natural or synthetic dyes [1,2]. Synthetic dyes are commonly used in most textile industries, replacing natural dyes due to their production cost and easy utilisation. However, synthetic dyes, such as azo dye, have had detrimental effects on the environment because of their toxicity, which can lead to skin

rashes and allergies. Some reports even claim they are mutagenic and carcinogenic [3]. As a result of high demand, this industry consumes a massive volume of water and releases effluent that contains a high load of contaminants into the water bodies, which can harm the surrounding aquatic and soil ecosystems [4–7]. Therefore, the Department of Environment, Malaysia (DOE) laws regarding removing dyes from textile industry effluents are becoming stricter in many developed and industrialized countries, such as Malaysia and Singapore, as it has been the primary source of water pollution [8]. Over the years, dye removal from textile and apparel industry wastewater has become a serious concern. Many textile industries favour conventional treatment by utilising chemicals due to the cost of high consumption [9].

Treating textile wastewater involves several stages before it can be released into a reservoir or collection area [10]. Environmental protection may be achieved using wastewater treatments such as membrane filtration, adsorption, and coagulation methods. Adsorption technology is a viable treatment method with a more straightforward design and less economical processing cost compared to existing wastewater treatment methods employed by the industry, which have issues such as unwanted fouling and higher cost maintenance [11]. It also effectively removes organic contaminants and heavy metals from wastewater. However, it has several limitations, including regenerating the adsorbent, because some adsorbents can only be used once [12]. In the industrial sector, conventional or inorganic adsorbents, including activated carbon, alumina and bentonites, are frequently used commercially [13].

Dolomite is a common rock-forming mineral found in sedimentary beds and is a generally accessible adsorbent as it has alternative layers of calcium oxide (CaO), magnesium oxide (MgO) and carbon dioxide (CO₂) [14]. It has been used in the float glass and fertiliser industries and has construction applications for soil conditioning and to prepare roads before paving [15]. In Peninsular Malaysia, dolomite is a mineral widely distributed in mountainous and cave regions. It is also common in northern Malaysia [16,17]. It is soft and can be easily ground into a fine powder. Greyish and yellowish colours are frequently recognized [18].

Recently, some studies showed that dolomite possesses adsorption capability due to its surface area and composition of calcite and magnesite, which allow it to immobilise metals and pH-neutralising agents in wastewater [19,20]. The adsorption properties of raw dolomite, such as specific surface area and pore volume, can be improved by thermal treatment of the material through partial calcination [21]. Additionally, this process increases the presence of functional groups on the surface of the dolomite, such as hydroxyl (OH), carboxyl (COOH) and metal ions, which may affect the pH of the effluent [22]. However, dolomite waste can be recycled using a desorption technique to stop pollutants from being unintentionally leached. Some studies have shown the low possibility of a leaching process in dolomite [23]. While research on synthetic textile dye wastewater has been conducted recently, real textile dye wastewater applications could be more frequently studied. Moreover, a study was performed on microbes' inhibition in wastewater due to pH alteration. This study investigated the optimal temperature for dolomite calcination for a better adsorption rate of colour and COD in the dye wastewater.

2. Materials and Methods

2.1. Activation of Dolomite

Raw dolomite samples from GMZ Agro Company were taken and sieved to sort for the necessary sizes (1.0 mm, 600 µm and 90 µm). The furnace was prepared to pre-heat until it reached the optimal temperatures (300 °C, 700 °C and 900 °C). The raw dolomite was placed in crucibles and heated in the furnace for 5 h. This amount of treatment time was sufficient for the calcination of dolomite. Dolomite undergoes two stages of thermal degradation: (1) the first phase is the formation of calcite (CaCO₃) and periclase (MgO), and (2) the second phase releases calcium oxide (CaO) because of the decay of CaCO₃.

2.2. Preparation of Textile Dye Wastewater

The RTDW appeared maroon-brownish and was received from the textile industry in Batu Pahat, Johor, Malaysia. In contrast, methyl orange, C.I.13025, ($C_{14}H_{14}N_3NaO_3S$, $327.34 \text{ g mol}^{-1}$, MQ200 grade) purchased from Sigma Aldrich, St. Louis, MO, USA, was used to produce the STDW. The initial absorbance reading for RTDW was 0.266 AU, while for STDW, the reading was 0.385 AU. To prepare the stock solution of STDW, an appropriate amount of methyl orange was dissolved in distilled water to the concentration of 100 mg/L. Each wastewater's pH was measured and recorded. To ensure the growth of the culture for STDW, 1 mL serial diluted (10^{-5}) *E. coli* was added to the solution using a micropipette, and the solution was left for three days at room temperature. Gram staining was applied to both samples to identify and observe the bacteria in the dyes.

2.3. Experimental Treatment

The treatment process involved identical procedures for implementing specific parameters using the Response Surface Methodology (RSM). For RTDW and STDW, the treatment was prepared in triplicate, and the requisite dosages of activated dolomite were added to the 100 mL wastewater sample, which was well stirred at a speed of 100 rpm [24]. The treatment was conducted over two hours, with another sedimentation interval before analysis. Amounts of 200 mL of RTDW and STDW (methyl orange) with concentrations of 100 mg/L without introducing dolomite were taken as control treatments. The variables that were examined were the activation temperature of the dolomite (300 °C, 700 °C and 900 °C), the size of the activated dolomite (1.0 mm, 600 μm and 90 μm), and the concentration of the absorbent employed (1.0 g, 3.0 g and 5.0 g). A summary of the applied methods is shown in Figure 1.

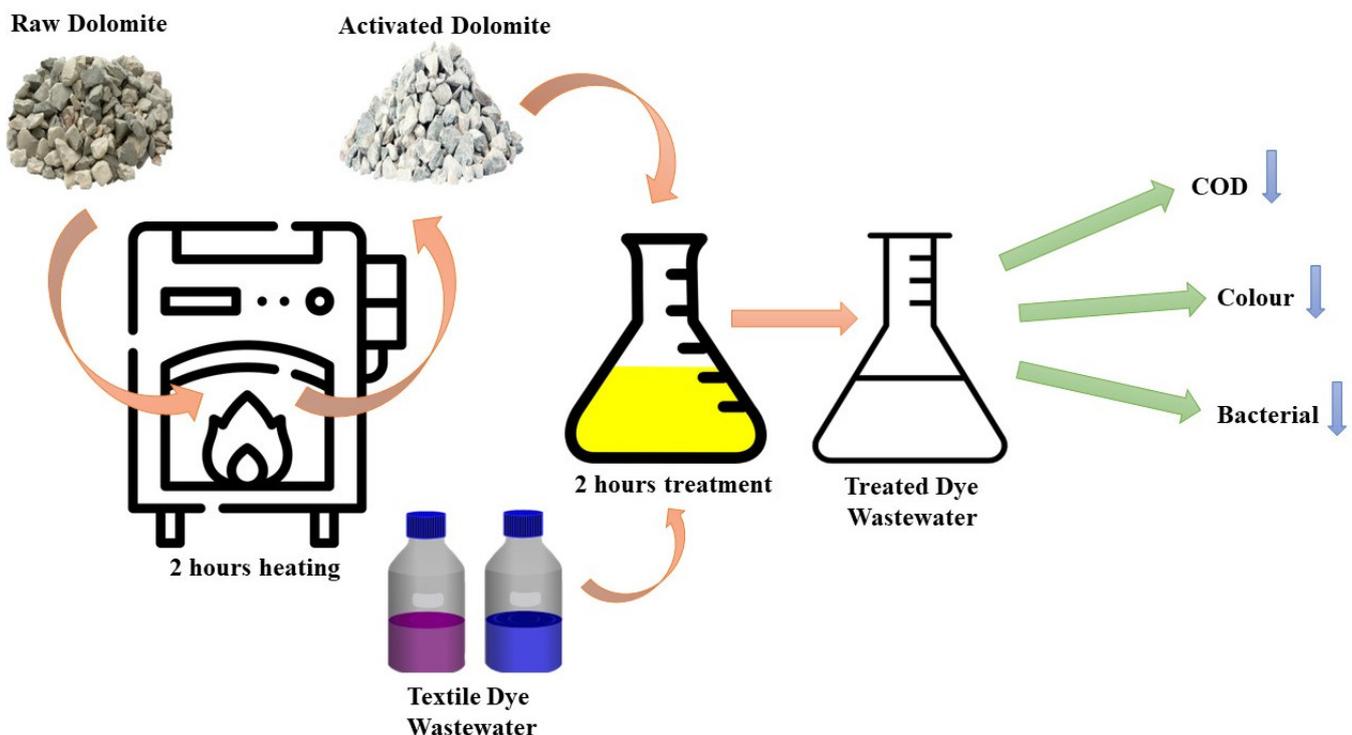


Figure 1. Graphical methodology of the treatment.

2.4. Morphological Analysis

Observation was conducted on pre- and post-treatment activated dolomite. The structures of the raw and activated dolomite were observed macroscopically and microscopically. The macroscopic view was performed under a dissecting microscope (Olympus Nikon

Microscope), and the microscopic image was under a Field Emission Scanning Electron Microscopy (FESEM) Model SUPRA 55VP (CARL ZEISS, Dresden, Germany).

2.5. Removal of Colour, COD and Adsorption Study

Each sample was drawn using a dropper for the decolourisation assay, transferred into a cuvette and then tested using UV-visible spectrophotometry (Shimadzu UV Mini 1240 Europe, Duisburg, Germany). The RTDW and STDW samples' absorbance wavelengths were 420 nm and 464 nm, respectively [24]. The adsorbed sum was determined by the difference between the beginning and end values. The percentage of decolourisation was then determined using Equation (1):

$$\text{Decolourisation (\%)} = \frac{(Abs_o - Abs_t)}{Abs_o} \times 100 \quad (1)$$

where the percentage of decolourisation was observed based on the difference between the average of initial absorbance (Abs_o) and the average of final absorbance over time (Abs_t).

In measuring chemical oxygen demand (COD), 2 mL of the extracted sample was added to COD Digestion Vials, High range (20 to 1500 mg/L) (standard protocol from the manufacturer; HACH, Kuala Lumpur, Malaysia) and incubated at 150 °C for 2 h. The vials were then measured in a DR6000 UV-VIS Laboratory Spectrophotometer (HACH, Kuala Lumpur, Malaysia) to obtain a concentration reading in mg/L. The percentage of COD reduction was indicated using Equation (2):

$$\text{COD Reduction (\%)} = \frac{(C_o - C_t)}{C_o} \times 100 \quad (2)$$

where C_o is the initial concentration of COD, and C_t is the temporal concentration of COD. The colour uptake per gram of sorbent was calculated according to Equation (3):

$$q_e = \frac{C_o - C_e}{W} \quad (3)$$

where q_e is the equilibrium adsorption capacity (mg/g), C_o is the initial concentration (mg/L) of the colour solution and C_e is the equilibrium concentration (mg/L) of the colour solution.

The equilibrium adsorption of STDW and RTDW was studied as a function of initial concentration in the range of 100 mg/L and 10 mg/L, respectively. The Langmuir and Freundlich adsorption isotherms were used in this study. The linear equations of the Langmuir and Freundlich isotherms are represented in Equations (4) and (5), respectively:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{1}{q_m} C_e \quad (4)$$

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \quad (5)$$

These equations present an adsorption model where q_e (mg/g) is the amount of colour adsorbed per unit mass of adsorbent particles at equilibrium, C_e (mg/L) is the equilibrium concentration of the STDW or RTDW, K_L is the equilibrium constant (L/mg) related to the Langmuir isotherm constants, q_m is the maximum adsorption capacity (mg/g) and K_F and n are Freundlich isotherm constants related to the adsorption capacity and adsorption intensity, respectively [25]. Therefore, in a Langmuir isotherm, a plot of C_e/q_e versus C_e should be a straight line with a slope of $1/q_m$ and intercept as $1/K_L q_m$. Moreover, a plot of $\log q_e$ versus $\log C_e$ should be linear with a slope of $1/n$ and intercepts as $\log K_F$ under a Freundlich isotherm.

2.6. Kinetics Study

The kinetics of the adsorption of STDW and RTDW by activated dolomite is studied using pseudo-first-order and pseudo-second-order models. The pseudo-first-order (Lagergren's) model describes adsorption in solid–liquid systems based on the sorption capacity of solids [26] and is generally expressed as Equation (6). The study of sorption kinetics describes the effect of time on sorption; this rate controls the contact time of the adsorbate at a solid–liquid interface or aqueous solutions [27].

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{0.2303}t \quad (6)$$

The values of q_t and q_e are the amounts absorbed per mass of sorbent (mg/g) at any time and equilibrium, respectively, and k_1 is the rate constant of first-order sorption (min^{-1}). The straight-line plot of $\log(q_e - q_t)$ against t gives $\log(q_e)$ as intercept and a slope equal to $-k_1/2.303$. Hence, the amount of solute sorbed per gram of sorbent at equilibrium (q_e) and the first-order sorption rate constant (k_1) can be evaluated from the slope and the intercept.

The pseudo-second-order chemisorption kinetic equation [28] is expressed as Equation (7):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

where q_e and q_t are the sorption capacity at equilibrium and time t (mg/g), respectively, and k_2 is the rate constant of the pseudo-second-order sorption ($\text{g}/\text{mg}\cdot\text{min}$). The pseudo-second-order rate expression was applied for analysing chemisorption kinetics from liquid solutions [28]. Suppose the pseudo-second-order kinetics applies to the experimental data. In that case, the plot of t/q_t versus t should give a linear relationship from which q_e can be determined from the slope, and $k_2 q_e^2$ obtained from the intercept of the plot.

2.7. Bacterial Assay

The water samples were then examined for the presence of bacteria after the wastewater had been treated with activated dolomite. A dilution factor of 10 was used by mixing 1 mL of the sample with 9 mL of distilled water. Diluted samples of 0.05 mL were prepared on the nutrient agar using the spread plate technique and allowed to grow for a day at room temperature. The following day, the number of colonies grown on the plates was counted. The formula used to determine the colony-forming unit (CFU) is shown as Equation (8):

$$\text{CFU/mL} = \frac{\text{number of colonies} \times \text{total dilution factor}}{\text{volume of sample plated (mL)}} \quad (8)$$

3. Results

3.1. Physical and Morphological Observation

Figure 2 demonstrates a comparison of features. Activated dolomite, which has a more greyish-white colour from carbonisation, is significantly greyer than raw dolomite. An increase in pore density following heat treatment, essential to adsorption as the active site for the dye binding site, makes activated dolomite lighter in weight [29].

After carbonisation, the activated dolomite contains more significant concentrations of trace elements such as O, Ca, C and Mg that exhibit heat treatment that changes their physical and chemical properties as stated in Table 1. After the heat treatment, observing other elements, including Fe, Ti, Mg, Si and Al, is feasible. The elements' weight indicates the elements' concentration on the dolomite surface. The weight of oxygen and carbon elements increased after the heat treatment of the raw dolomite, while magnesium and calcium decreased.

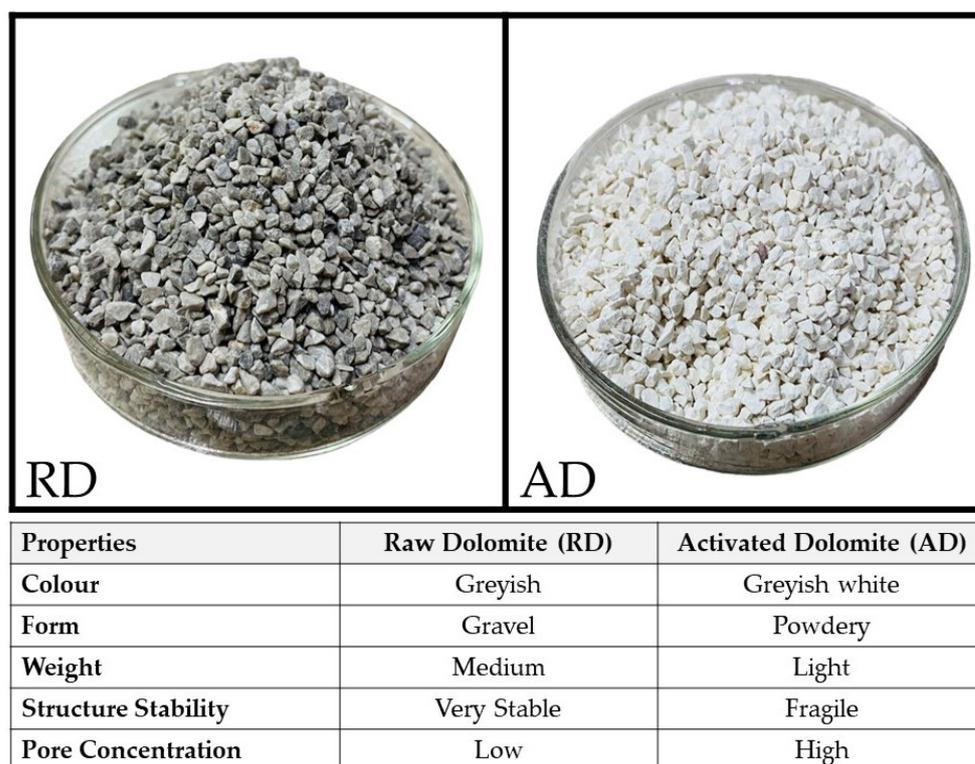


Figure 2. The comparison between raw dolomite (RD) and activated dolomite (AD) and their physical properties (down).

Table 1. The energy dispersive X-ray analysis (EDX) on raw dolomite (control), activated dolomite (control) and treated activated dolomite in RTDW and STDW treatment.

Raw Dolomite (Weight %)		Activated Dolomite (Weight %)		Treated Activated Dolomite (Weight %)	
O	33.47	O	41.17	O	42.27
Ca	38.73	Ca	24.13	Ca	27.6
C	6.7	C	16.13	C	13.93
Mg	21.13	Mg	14.4	Mg	14.3
		Fe	1.8	Fe	1.8
		Ti	0.9	Ti	0.8
		Mn	1.2	Mn	0.6
		Si	0.2	Si	0.2
		Al	0.1	Al	0.1

The presence of elements on the dye-treated dolomite exhibits a modest weight increase compared to the control dolomite, indicating that adsorption has occurred.

Dolomite is a physical adsorbent similar to any other activated carbon and gravel filtration compound. It can absorb the contaminants onto its surface, which is rich with pores. In this study, the observation of dolomite was conducted using a dissecting microscope (DM) and Field Emission Scanning Electron Microscopy (FESEM) to investigate and characterise the surface morphology and fundamental physical characteristics of the adsorbent surface, which was advantageous for mapping out the particle shape, porosity and size distribution of the activated dolomite. FESEM is a non-destructive form of electron microscopy in which the sample's surface is contacted by an electron beam to produce signals at extremely high magnifications in the nm range. The DM and FESEM images of dye-treated and untreated activated dolomite are displayed in Figure 3.

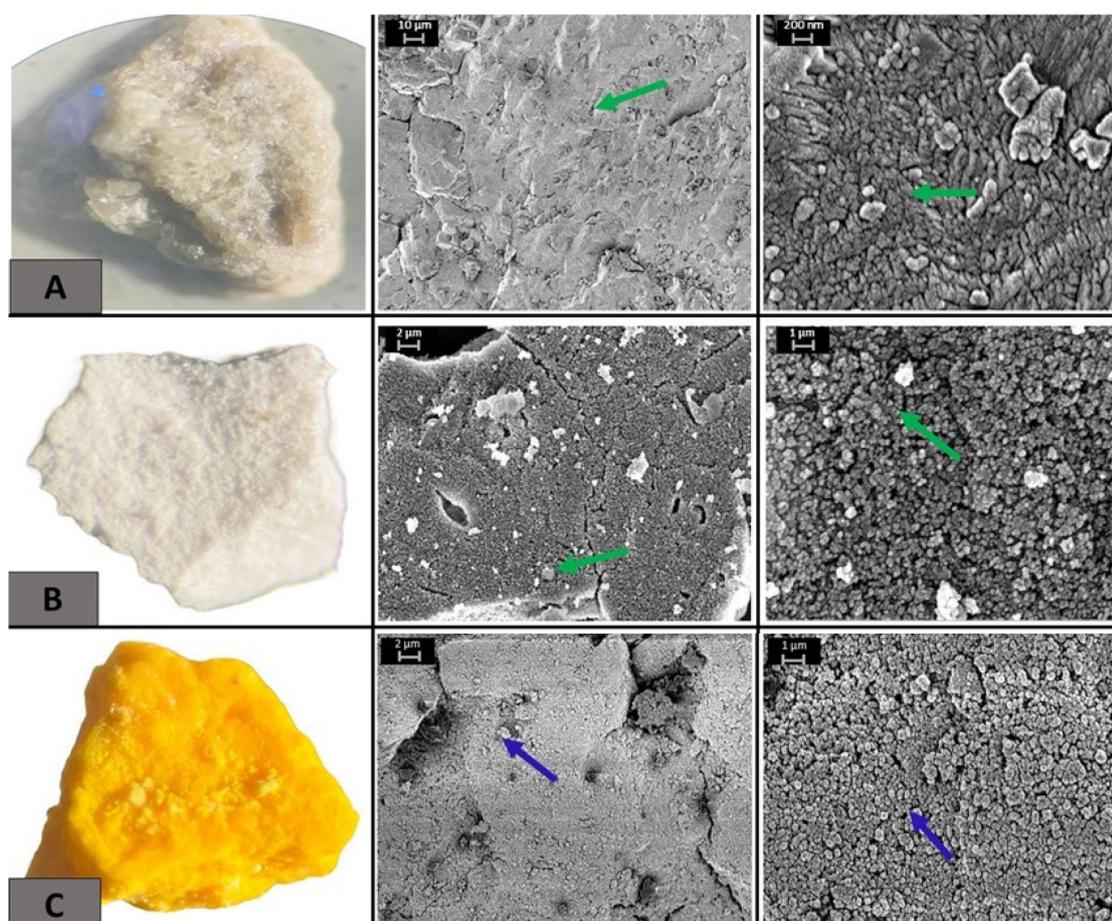


Figure 3. Raw and activated dolomite before and after being treated with RTDW and STDW using a dissecting microscope (DM—left picture) and Field Emission Scanning Electron Microscopy (FESEM—middle and right picture). Raw dolomite = inactivated dolomite. Bar (FESEM) = 10 μm and 200 nm (A); Control: untreated activated dolomite at 700 $^{\circ}\text{C}$. Bar (FESEM) = 1 μm and 2 μm (B); Activated dolomite treatment in RTDW and STDW. Bar (FESEM) = 1 μm and 2 μm (C). Blue arrows indicate dye compound adsorption on dolomite (dense clumping on pores). Green arrows indicate no dye compound adsorption.

SEM analysis evaluates the dolomite adsorption on the dye compounds (Figure 3). Figure 3 shows FESEM images of inactivated and activated dolomite. Raw dolomite is depicted in Figure 3A using SEM images, and the surface is very disordered with various-sized pores. Figure 3B, showing activated dolomite, exhibits smaller, more prominent pores that are evenly distributed; these display the reduction in dolomite due to carbonate decomposition and the thermal cracking of dolomite fragments.

When compared to raw dolomite and untreated activated dolomite as a control, activated dolomite exhibited dye adsorption (pores: green arrows in Figure 3A,B), which was followed by increasing densities of clumps on the pores, indicating that the dye molecules had coated the dye-adsorbed dolomite. The control dolomite in Figure 3A,B (left pictures) is grey, indicating that it is unsaturated, which is consistent with the findings of [17,27]. The blue arrows are in Figure 3C.

3.2. Optimisation of RTDW Decolourisation

Table 2 displays the ANOVA for RTDW dye decolourisation. The difference between the predicted R^2 (0.77) and adjusted R^2 (0.69) is less than 0.2, indicating reasonable agreement. The model p -value in Table 2 is <0.005 ($p < 0.0015$), indicating high-significance for the model research. Given that this model's precision is greater than 4.0, it is functional in

the design space. The significance of the model was derived from the regressed adjusted coefficient of determination ($\text{adj. } R^2 = 0.77$), as stated in Equation (9). The F-value of 8.14 for the model illustrates its applicability. The 0.42 lack of fit F-value shows that noise is likely to cause a significant lack of fit F-value in 81.8% of occurrences (not significant).

$$\text{Decolourisation Percentage} = -39.24468 + 0.308352 \times \text{Temperature} - 0.005340 \times \text{Size} + 3.39432 \times \text{Weight} + 0.000017 \times \text{Temperature} \times \text{Size} - 0.002987 \times \text{Temperature} \times \text{Weight} - 0.006232 \times \text{Size} \times \text{Weight} - 0.000204 \times (\text{Temperature})^2 + 0.000018 \times (\text{Size})^2 - 0.228076 \times (\text{Weight})^2 \quad (9)$$

Table 2. The experimental results from the CCD quadratic model for dye decolourisation of RTDW.

Source	Sum of Squares	df	Mean Square	F-Value	p-Value	
Model	6881.22	9	764.58	8.14	0.0015	significant
A-Temperature	3707.01	1	3707.01	39.47	<0.0001	
B-Size	77.91	1	77.91	0.8295	0.3839	
C-Weight	397.72	1	397.72	4.23	0.0666	
AB	44.25	1	44.25	0.4711	0.5081	
AC	26.26	1	26.26	0.2796	0.6085	
BC	258.05	1	258.05	2.75	0.1284	
A ²	708.70	1	708.70	7.54	0.0206	
B ²	38.29	1	38.29	0.4076	0.5375	
C ²	2.29	1	2.29	0.0244	0.8791	
Residual	939.31	10	93.93			
Lack of Fit	277.84	5	55.57	0.4200	0.8185	not significant
Pure Error	661.47	5	132.29			
Cor Total	7820.53	19				
Std. Dev.	9.69		R ²	0.8799	Adeq. Precision	9.8000
Mean	67.79		Adjusted R ²	0.7718		
C.V. %	14.30		Predicted R ²	0.6945		

Figure 4 includes 3-D graphs that depict the effects of temperature, size and weight. Each panel displays the two components at optimal levels while the other component’s experimental range remains constant. Figure 4A,C demonstrates that weight and adsorbent size were insignificant factors in dye decolourisation, whereas activation temperature was highly significant ($p = 0.0001$). Figure 4B shows how the weight of the adsorbents affected decolourisation typically, whereas the size of the adsorbents was planar since it did not significantly affect the process.

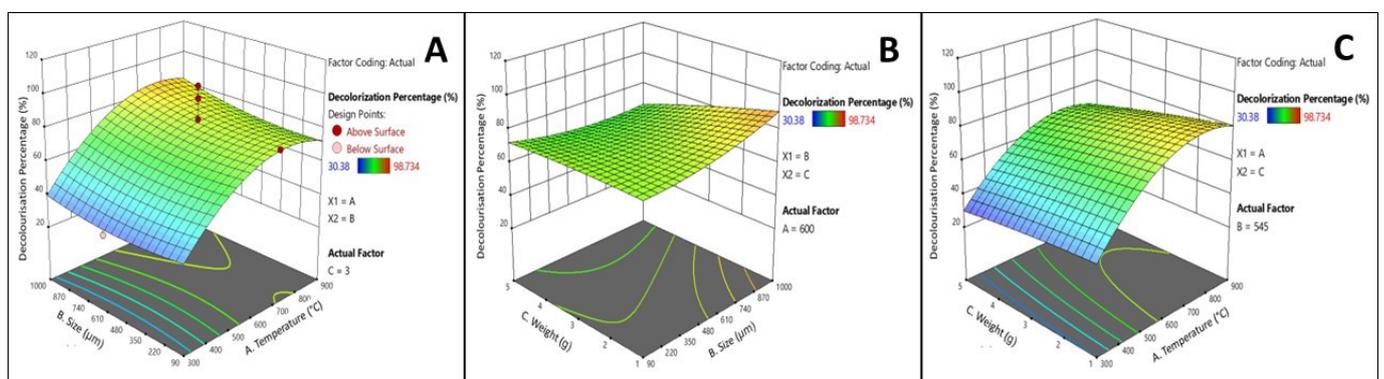


Figure 4. Response–surface curve (3-D plot) of dye decolourisation of RTDW showing the interaction between (A) temperature vs size, (B) size vs weight and (C) temperature vs weight.

3.3. Optimisation of STDW Decolourisation

Table 3 displays the ANOVA for STDW dye decolourisation. The difference between the predicted R² (0.76) and adjusted R² (0.56) is less than 0.2, indicating reasonable agree-

ment. The model *p*-value in Table 2 is <0.005 (*p* < 0.0018), indicating high-significance for the model research. Given that this model’s precision is greater than 4.0, it is functional in the design space. The significance of the model was derived from the regressed adjusted coefficient of determination (adj. *R*² = 0.76), as stated in Equation (10). The F-value of 7.75 for the model illustrates its applicability. The 0.54 lack of fit F-value shows that noise is likely to cause a significant lack of fit F-value in 73.7% of occurrences (not significant).

$$\begin{aligned} \text{Decolourisation Percentage} = & -0.927608 + 0.109659 \times \text{Temperature} + 0.041165 \times \text{Size} - 3.37479 \times \text{Weight} - \\ & 3.14484 \times 10^{-6} \times \text{Temperature} \times \text{Size} + 0.007034 \times \text{Temperature} \times \text{Weight} - 0.002174 \times \text{Size} \times \text{Weight} - \\ & 0.000063 \times (\text{Temperature})^2 - 0.000025 \times (\text{Size})^2 + 0.377027 \times (\text{Weight})^2 \end{aligned} \quad (10)$$

Table 3. The experimental results from the CCD quadratic model for dye decolourisation of STDW.

Source	Sum of Squares	df	Mean Square	F-Value	<i>p</i> -Value	
Model	3917.47	9	435.27	7.75	0.0018	significant
A-Temperature	2625.57	1	2625.57	46.74	<0.0001	
B-Size	54.77	1	54.77	0.9750	0.3467	
C-Weight	147.05	1	147.05	2.62	0.1367	
AB	1.51	1	1.51	0.0269	0.8730	
AC	145.66	1	145.66	2.59	0.1384	
BC	31.39	1	31.39	0.5588	0.4720	
A ²	66.73	1	66.73	1.19	0.3013	
B ²	73.08	1	73.08	1.30	0.2806	
C ²	6.25	1	6.25	0.1113	0.7455	
Residual	561.72	10	56.17			
Lack of Fit	198.98	5	39.80	0.5486	0.7371	not significant
Pure Error	362.74	5	72.55			
Cor Total	4479.19	19				
Std. Dev.	7.49		<i>R</i> ²	0.8746	Adeq. Precision	8.4772
Mean	56.43		Adjusted <i>R</i> ²	0.7617		
C.V. %	13.28		Predicted <i>R</i> ²	0.5625		

Figure 5 shows 3-D graphs exhibiting the effects of temperature, size and weight. Each panel depicts the two factors at their optimal levels while keeping the experimental range of the third factor constant. Figure 5A,C demonstrates that, in the dye decolourisation process, the activation temperature was very significant (*p* = 0.0001), while the other parameters were only marginally significant. Figure 5B demonstrates how the adsorbents’ weight minimally impacted decolourisation, whereas the adsorbents’ size was planar because it had no appreciable influence on the process.

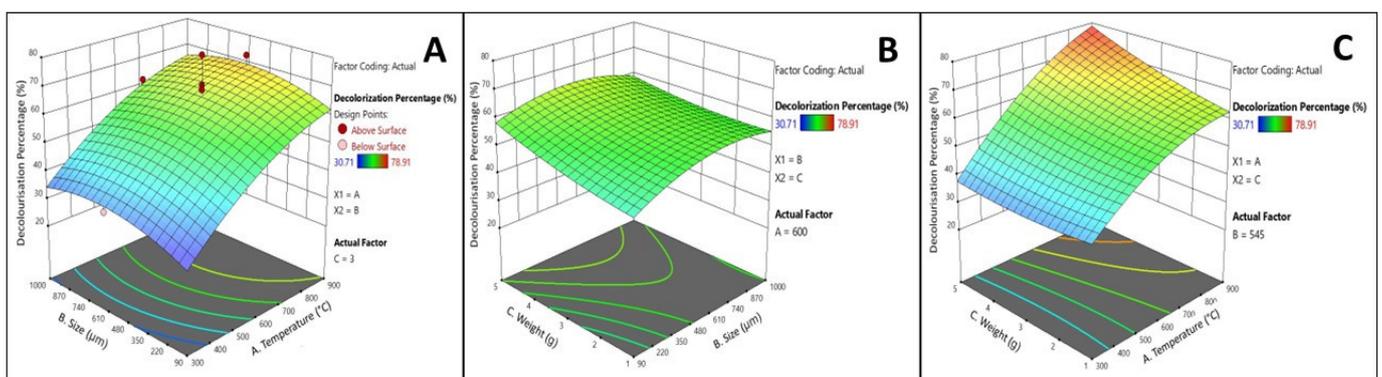


Figure 5. Response–surface curve (3-D plot) of dye decolourisation of STDW showing the interaction between (A) temperature vs size, (B) size vs weight and (C) temperature vs weight.

3.4. Effect of Activation Temperature

The ideal working performance of activated dolomite for dye removal was first described using three different activation temperatures for a 2-h treatment. The results were discussed based on the percentage of colour removed and COD reduction from the RTDW and STDW. The activated dolomite adsorption technique removes the colour from the textile dye effluent. Like other activated carbon adsorbents, the activated dolomite's surface complexation and ion exchange caused the mechanism [30]. The dye ions' removal from the wastewater depends on the volume of precipitation of their hydroxides formed in the presence of dolomite and the dissolution of magnesium and calcium carbonates from the dolomite lattice [31].

3.4.1. Colour Percentage Removal

The effectiveness of activated dolomite in decolourising the RTDW and STDW in the bioreactor is significantly influenced by the activation temperature of the dolomite. Figure 6 (left) indicates that the different temperatures at which the dolomite was activated substantially impacted the dye adsorption percentage removal during the treatment when the agitation speed was kept constant at 100 rpm. According to the results, the maximum percentage of the contaminant was removed within 2 h of treatment at 700 °C and 900 °C.

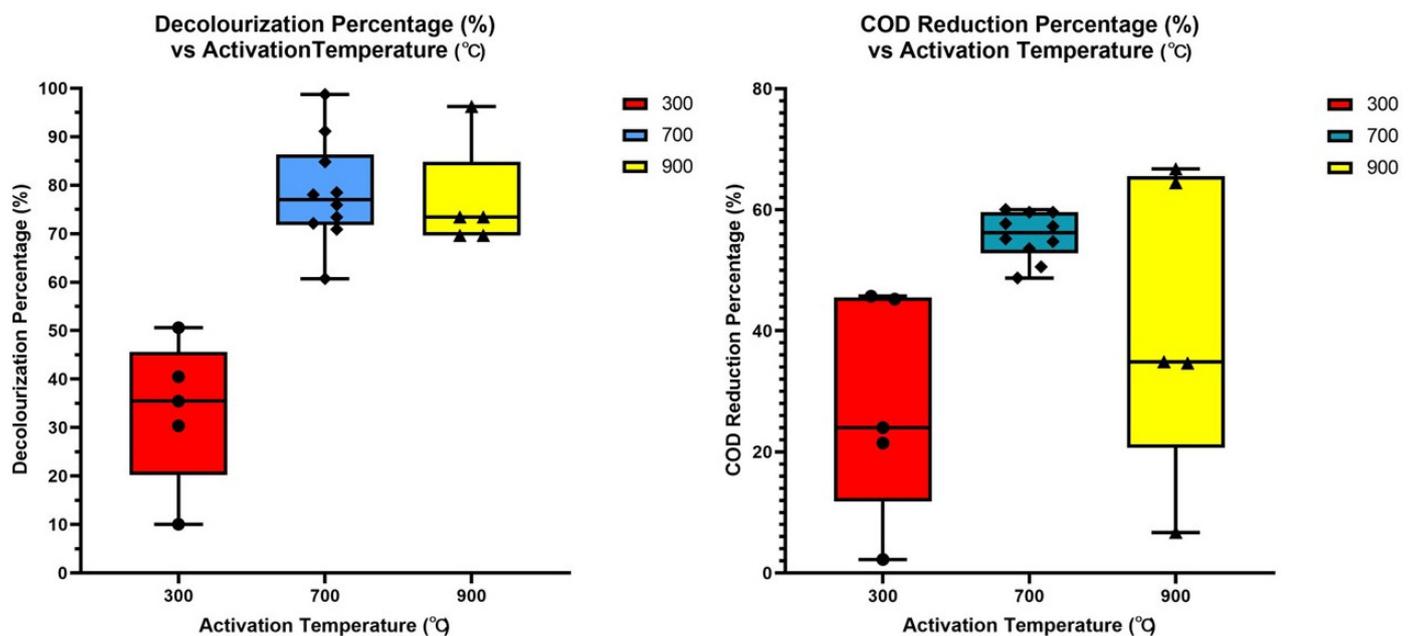


Figure 6. The decolourisation percentages (left) and COD reduction percentages (right) of RTDW based on activation temperature.

For dye decolourisation of RDTW, the maximum percentage removal was reached at 98.7%, and 96.2% for 700 °C and 900 °C, respectively, and the average decolourisation was within the range of 70.0% to 80.0% in most of the treatments. The dye decolourisation decreased significantly when the activation temperature was dropped to 300 °C, at which temperature only 10.0% of the colour was removed via the treatment. In the STDW treatment (Figure 7 (left)), the maximum decolourisation of dye by the activated dolomite was around 78.0% at a temperature of 700 °C. However, the treatment succeeded in decolourising the dye by more than 30.0% after 2 h, and the average decolourisation percentage was between 40.0% and 60.0%.

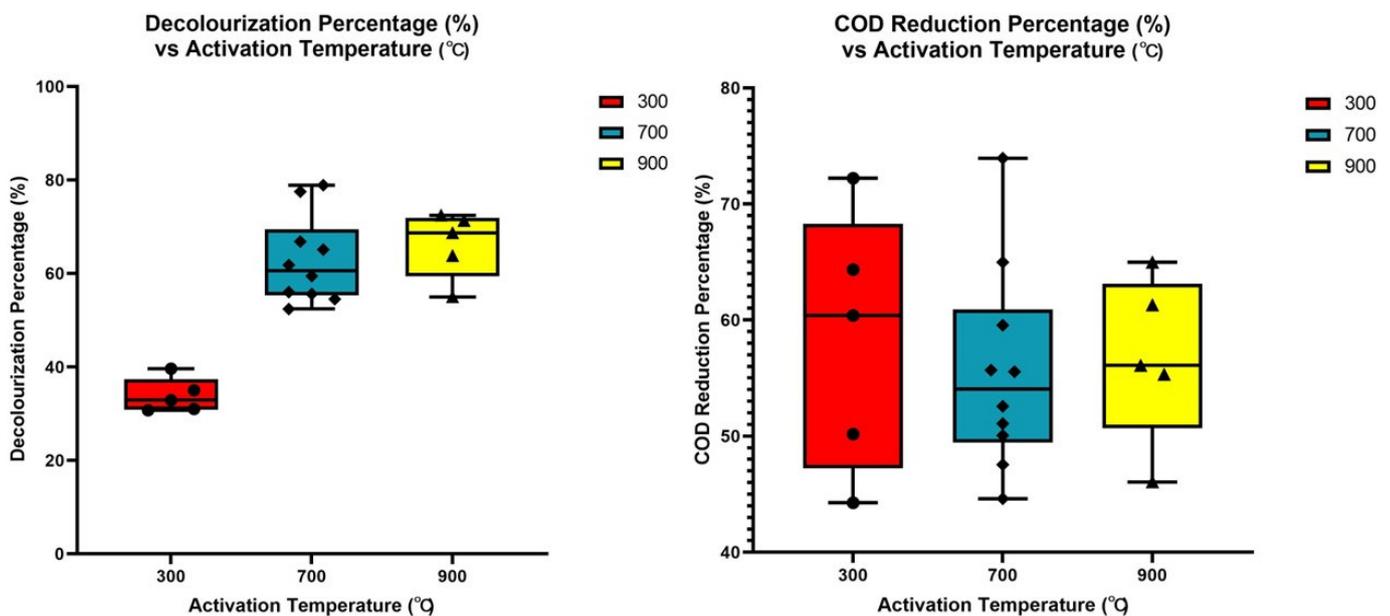


Figure 7. The decolourisation percentages (left) and COD reduction percentages (right) of STDW based on activation temperature.

3.4.2. COD Percentage Reduction

In Figures 6 and 7 (right), the RTDW and STDW were analysed for chemical oxygen demand (COD) to ascertain the affinity of the adsorbent for different compounds present in textile effluent. The 900 °C-activated dolomite provided the maximum removal of COD, at 69.0% as indicated by the data, verified by the triplicated studies performed on each sample. Significant discrepancies were shown in the samples treated by 300 °C- and 900 °C-activated dolomite as the COD reduction was inconsistent for each treatment. As for STDW, the COD reduction percentage showed average trends within the range of 50.0% to 65.0% for each activation energy.

3.5. Effect of Adsorbent Size

Previous analysis has shown that the activation temperature impacted the effectiveness of activated dolomite in treating RTDW and STDW. The following characteristics would apply to the optimal activation temperature of 700 °C. The size of activated dolomite was chosen to study its capacity better. To represent the typical size of dolomite in nature and its impact on the decolourising performance process in various sizes, dolomite samples with sizes between 90 and 1000 µm were employed.

3.5.1. Colour Percentage Removal

Figures 8 and 9 (left) depict that the size of the activated dolomite adsorbent utilised in this investigation had a considerable impact on the removal of colour. In the RTDW treatment (Figure 8 (left)), the 600 µm size had the maximum colour removal (98.7%), followed by 1000 µm (96.2%), while the average decolourisation percentage was 70.0% for every size. The results are similar to the STDW treatment in Figure 9 (left), as 600 µm had the highest colour removal percentage (78.0%). However, the sizes utilised in the treatment are comparable since they can adsorb the dye onto their surface and because they are chosen in small particles, which generally have a higher rate of dissolution due to their larger surface area to volume ratio and, therefore, a better possibility for faster absorption.

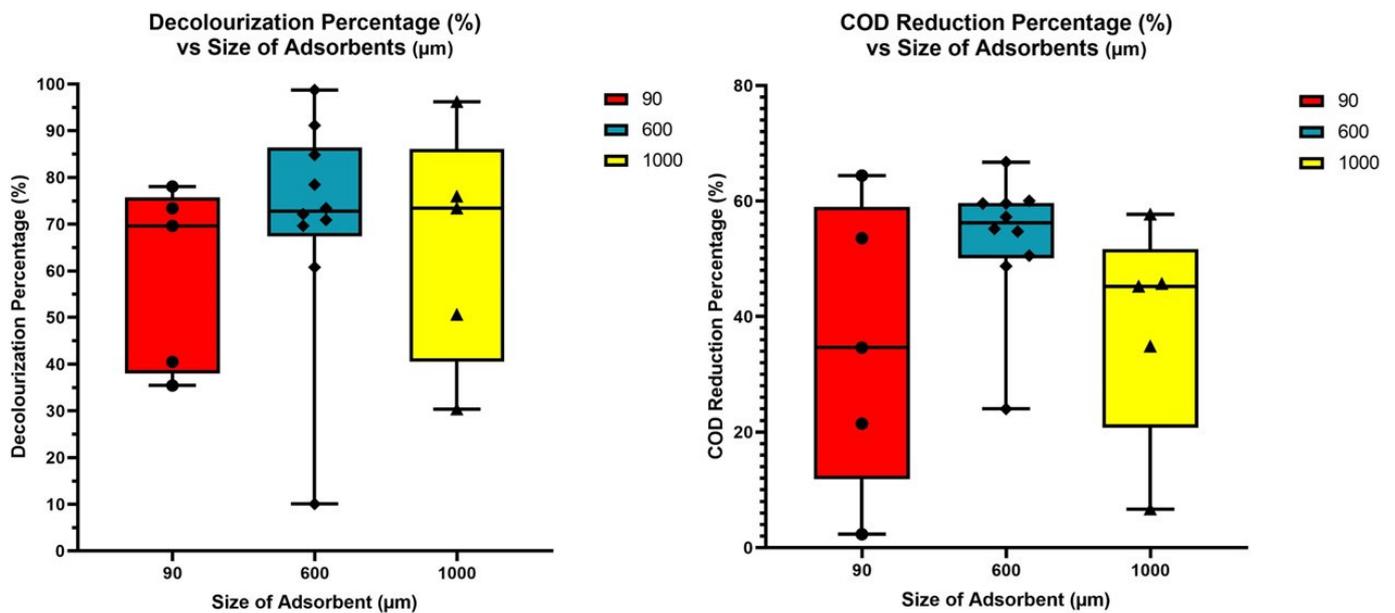


Figure 8. The decolourisation percentages (left) and COD reduction percentages (right) of RTDW based on the size of adsorbents in µm.

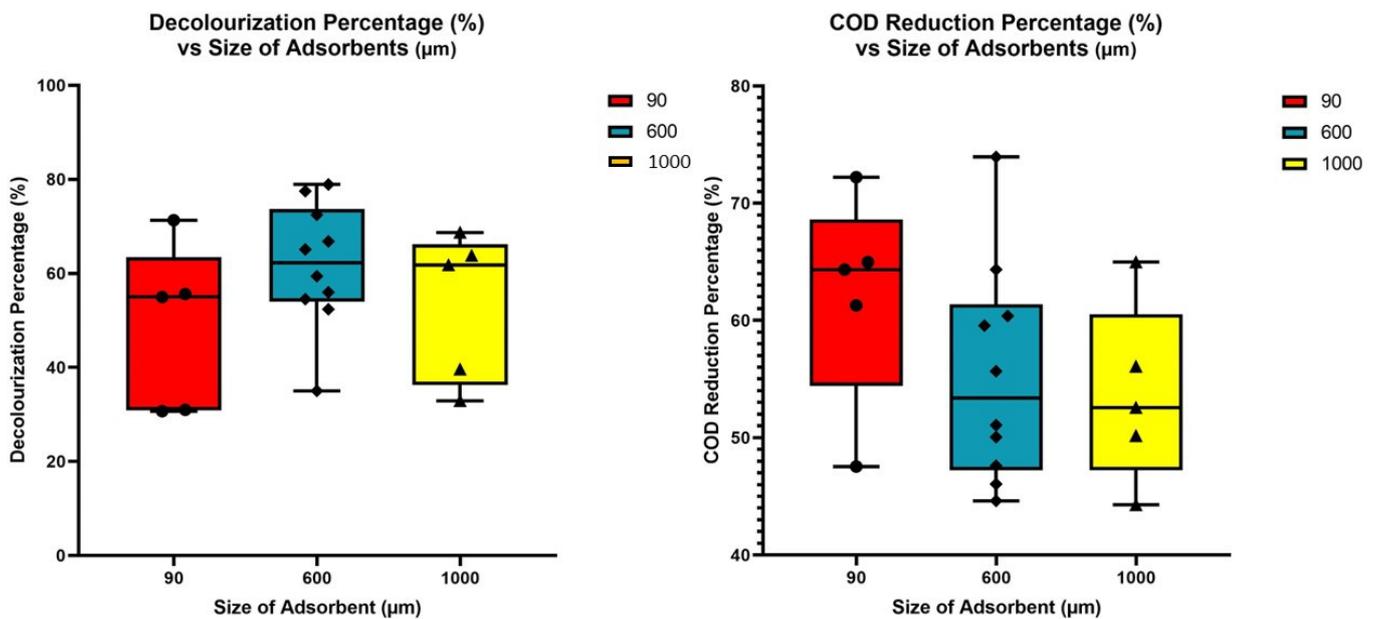


Figure 9. The decolourisation percentages (left) and COD reduction percentages (right) of STDW based on the size of adsorbents in µm.

3.5.2. COD Percentage Reduction

Despite variations in the number of adsorption sites and surface area of the activated dolomite, the COD reduction had the same adsorption kinetics as colour removal. The effect of adsorbent size on COD reduction is shown in Figure 8 (right). For a two-hour treatment using powdered activated dolomite, the percentages of COD reduction were 66.7% (600 µm) and 64.4% (90 µm). The highest percentage, obtained for the 1000 µm size, was 57.4%. In Figure 9 (right), the maximum COD reduction percentage in STDW is 74.5%, followed by 72.8%. The range of COD reduction percentages for RTDW and STDW treatments is nearly the same, ranging from 20.0% to about 60.0% to 70.0%.

3.6. Effect of Adsorbent Concentration

As well as determining the size of the dolomite and the influence of activation temperature, the concentration of adsorbents in the treatment was examined. Even though dolomite has been relatively non-toxic to the environment, it is a physical substance that cannot be easily broken down. The experiment was based on a concentration from a prior study [32], which found that when activated dolomite concentrations were 50 mg/L, it was possible to assess their efficacy. Additionally, the author conducted a preliminary experiment in which dye water was treated with 0.5 g of activated dolomite. Therefore, constant concentrations of activated dolomite of 1 g, 3 g and 5 g in the bioreactor at an agitation speed of 100 rpm and room temperature are recommended to achieve the best results.

3.6.1. Colour Percentage Removal

Figure 10 (left) shows that even at concentrations as low as one percent, the colour from the treatment can be effectively removed. In RTDW treatment, the percentage of colour removal can exceed 99.0% in higher adsorbent concentrations of one percent, three percent and five percent. Due to different activation temperatures during the preparation of the activated dolomite that could have impacted the treatment, some treatments did not provide favourable results despite employing the same concentration (three percent).

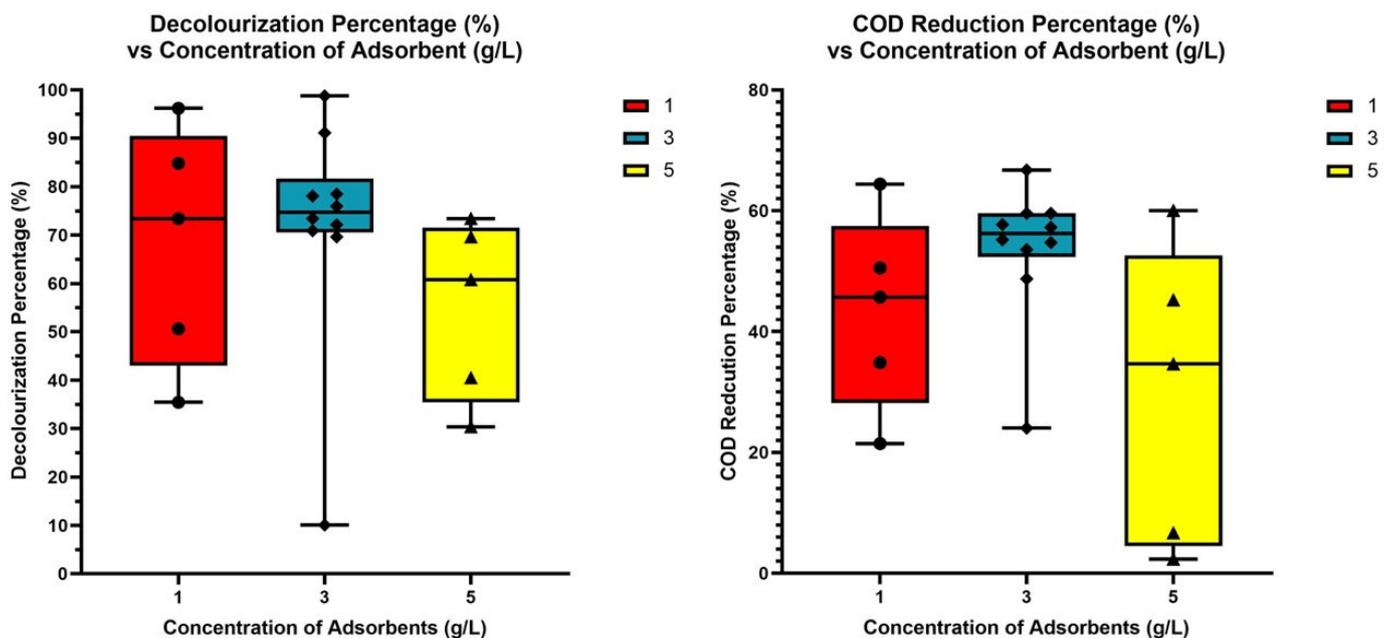


Figure 10. The decolourisation percentages (left) and COD reduction percentages (right) of RTDW based on the concentration of adsorbents.

In contrast to STDW treatment, RTDW treatment (Figure 10 (left)) can remove more than 50.0% of the colour from the samples with a lower proportion of the colour loss. The overall colour reduction assay results demonstrated how significant the dolomite's activation temperature is to enhance adsorption because it exhibits significant differences from other parameters.

3.6.2. COD Percentage Reduction

The COD reduction from the samples can be determined with the same concentration of adsorbents. The concentrations of adsorbents employed in RTDW (Figure 10 (right)) treatment produced approximately the same pattern in colour removal, with three percent having the highest point of COD reduction. In contrast, other percentages were still capable

of providing the reduction. However, in STDW treatments (Figure 11 (right)), the average COD reduction was between 50.0% and 60.0% for every concentration. Overall, the COD reduction assay on both dye wastewaters showed significant discrepancies between each factor's lowest and highest points.

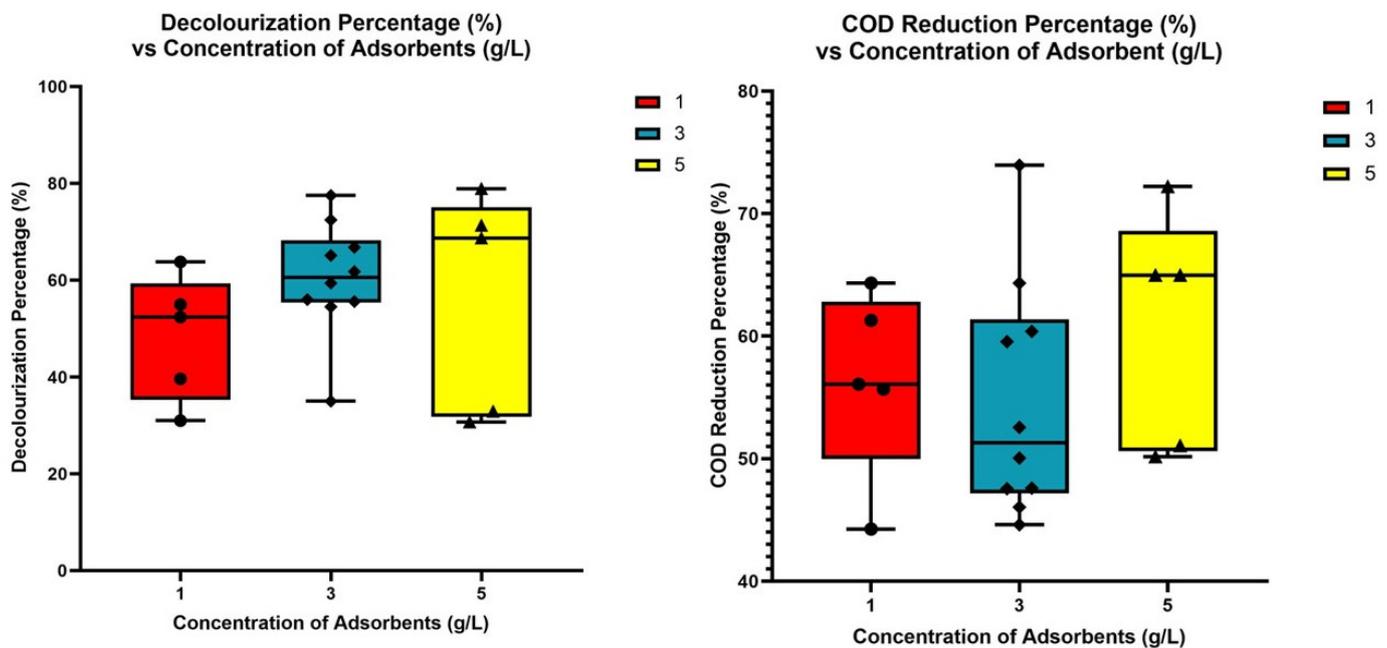


Figure 11. The decolourisation percentages (left) and COD reduction percentages (right) of STDW based on the concentration of adsorbents.

3.7. Adsorption Isotherms

This study was conducted to determine the equilibrium adsorption of the dolomite when treating RTDW and STDW based on the contact time. As shown in Figure 12, it was observed that the rate of colour uptake for RTDW increased rapidly in the first 60 min of contact time. After about 60 min, the rate decreased until a constant colour concentration was reached; this occurred after about 60 min for RTDW. This time was presumed to represent the equilibrium time at which the equilibrium metal concentration occurred. For STDW, slightly unstable adsorption was observed after 80 min, showing some detachable colour from the dolomite surface. The assumption was made that after 80 min, the equilibrium was achieved.

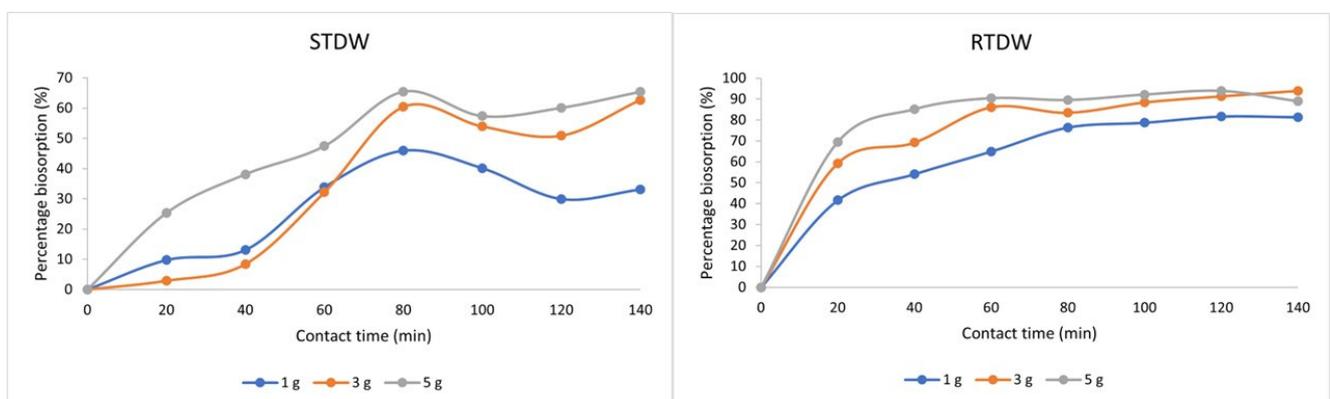


Figure 12. STDW and RTDW percentage biosorption onto activated dolomite against time. (Experimental conditions: C_o STDW = 100 mg/L; C_o RTDW = ~10.0 mg/L; adsorbent dosage = 1.0 g, 3.0 g and 5.0 g per 100 mL, dolomite T = 700 °C, particle size > 1.0 mm, mixing rate = 100 rpm, T = 25 ± 1 °C).

3.7.1. Study of Adsorption Model

For the adsorption model and kinetics, one mass of activated dolomite was chosen to represent the study and the obtained data from the linear isotherm in Table 4. The data of the adsorption isotherms model for STDW and RTDW show that the adsorptive behaviour of colour on activated dolomite satisfies the Langmuir assumptions following the R^2 values ($R^2_{STDW} = 0.9484$ and $R^2_{RTDW} = 0.9686$). The maximum adsorption capacities for STDW and RTDW were 0.3931 and 0.3519 mg/g, respectively. This result showed that the STDW and RTDW are homogeneously adsorbed on the surface of the dolomite.

Table 4. Langmuir and Freundlich isotherm constants for adsorption of STDW and RTDW. (Experimental conditions: C_0 STDW = 100 mg/L; C_0 RTDW = 10.0 mg/L; adsorbent dosage = 1.0 g per 100 mL, dolomite T = 700 °C, particle size > 1.0 mm, mixing rate = 100 rpm, T = 25 ± 1 °C).

Adsorbates	Langmuir Isotherm			Freundlich Isotherm		
	q_m (mg/g)	K_L (L/mg)	R^2	K_F	n	R^2
STDW	0.3482	0.0203	0.9484	338.49	−0.3253	0.9383
RTDW	0.3519	0.8490	0.9686	1.0603	−1.8501	0.9493

3.7.2. Study on Adsorption Kinetics

From the obtained kinetic rate constants, the pseudo-first- (k_1) and pseudo-second-order (k_2) models of sorption of STDW and RTDW on the activated dolomite were calculated, as shown in Table 5. The fitted equilibrium adsorption capacities derived from Equation for RTDW are similar to those observed experimentally, which were 0.8984 mg/g and 0.8719 mg/g for both the pseudo-first-order model and pseudo-second-order. Contrarily, for STDW, the result for the kinetic model was higher than the experimental values of 6.0076 mg/g and 4.1946 mg/g for the pseudo-first-order and pseudo-second-order, respectively.

Table 5. Pseudo-first-order and pseudo-second-order data for the adsorption of STDW and RTDW. (Experimental conditions: C_0 STDW = 100 mg/L; C_0 RTDW = 10.0 mg/L; adsorbent dosage = 1.0 g per 100 mL, dolomite T = 700 °C, particle size > 1.0 mm, mixing rate = 100 rpm, T = 25 ± 1 °C).

Adsorbates	q_{exp} (mg/g)	Pseudo-First-Order			Pseudo-Second-Order		
		K_1 (min ^{−1})	q_e (mg/g)	R^2	K_2 (g/mg·min)	q_e (mg/g)	R^2
STDW	2.60	0.0044	6.0076	0.5685	0.00537	4.1946	0.5137
RTDW	0.80	0.0221	0.8984	0.9879	0.07475	0.8719	0.9592

The pseudo-first-order model best describes STDW because it has a higher correlation coefficient than the pseudo-second-order model. The correlation coefficients and R^2 values for the pseudo-first-order and pseudo-second-order models for RTDW are 0.9879 and 0.9592, respectively. Given the excellent agreement between model fit and experimentally observed equilibrium adsorption capacity and the significant correlation coefficients, this suggests that RTDW adsorption followed pseudo-second-order kinetics and was adsorbed onto dolomite via chemical interaction.

3.8. Antimicrobial Properties

After the treatments were concluded as shown in Figure 13, 1 mL of the sample was removed, poured on nutrient agar and left to incubate for 24 h. The initial bacterial count on the plate was 53,400 CFU/mL, and the treated plate was monitored over the following 24 h. According to Figure 14, most treatments resulted in minimal microbial growth in RTDW and STDW treatments compared to the initial samples. The highest percentage of bacterial growth inhibition observed in RTDW and STDW was 99.7% and

98.6%, respectively, and the lowest percentage of bacterial growth inhibition in RTDW and STDW was 62.1% and 66.7%.

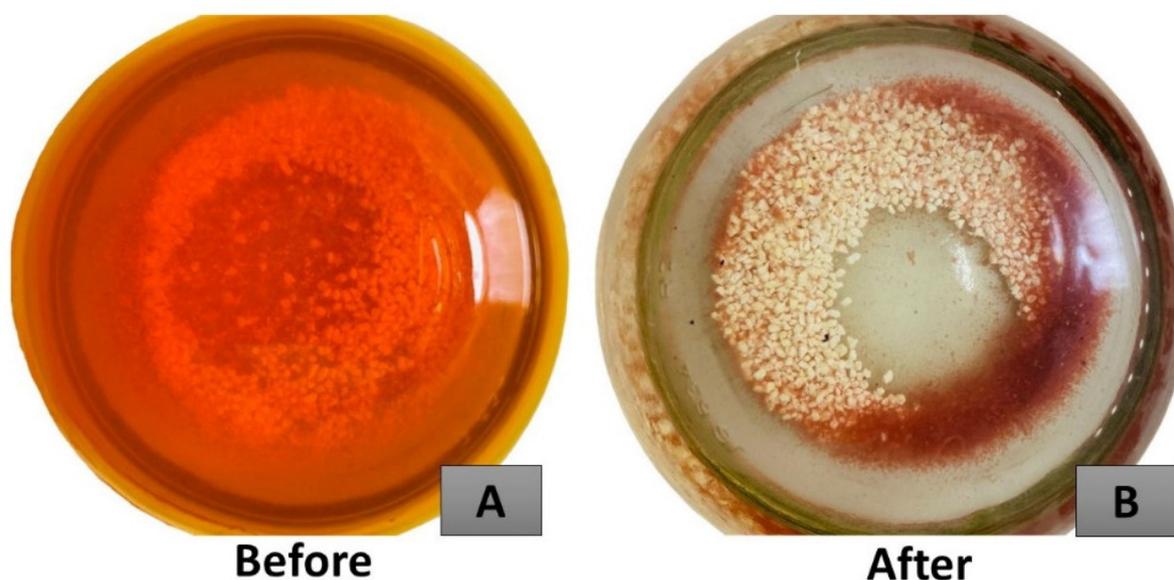


Figure 13. The textile dye wastewater before treatment (A) and after treatment (B).

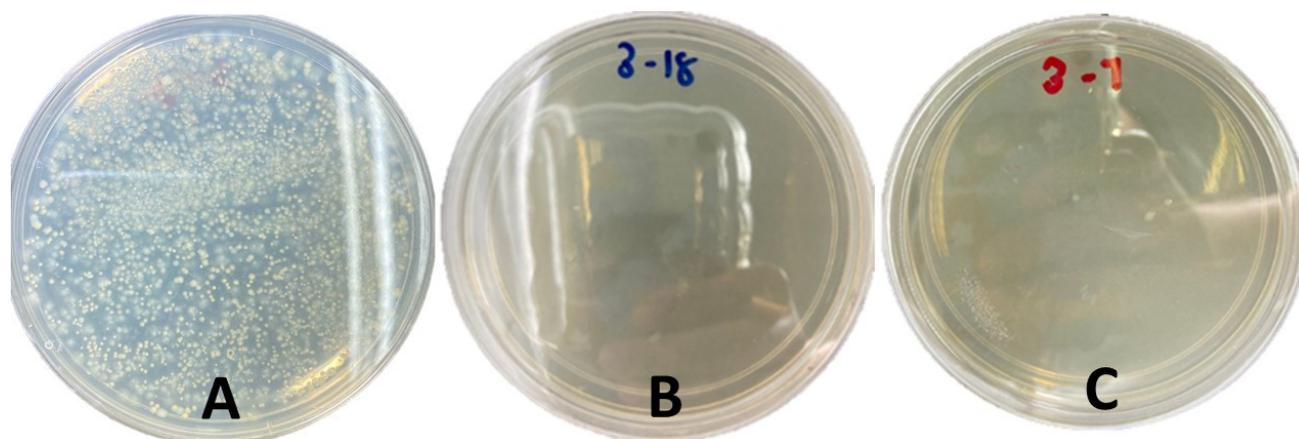


Figure 14. Antimicrobial analysis on nutrient agar (NA). Control (A), RTDW (B) and STDW (C). The samples were incubated for 24 h at room temperature.

3.9. Comparison of Current Work on Adsorption Using Activated Dolomite with Other Types of Adsorbents

The adsorption of dye using dolomite was compared with other adsorbents, as shown in Table 6. The activation temperature of dolomite used in this study is similar to Ziane [24], Khalizadeh [30], Walker [31] and within the range of 700 °C to 900 °C, and the retention time on the decolourisation was similarly within 2 h. However, the colour reduction obtained was higher than the other studies: 98.70% and 78.0% within 2 h. The study by Birniwa [29] on adsorption by Polypyrrole-Polyethyleneimine nano adsorbents on removing methylene blue demonstrated that adsorption reached 99% within 24 h. The study conducted by Kutty [33] reported on adsorption using activated sludge plants with 95% colour reduction from the treated palm oil mill effluents. The current study also reported COD and microbial reduction, which were not reported in any of the other studies.

Table 6. Comparison of current works on adsorption of dye using activated dolomite and other adsorbents.

Type of Adsorbents	Compound Removed	Activation Temperature	Retention Time	COD Reduction	Colour Reduction	Microbial Reduction	Ref.
Activated Dolomite	Textile and Methyl Orange	300 °C, 700 °C, 900 °C	2 h	66.70%, 73.95%	98.70%, 78.0%	Yes	This study
Activated dolomite	Reactive Black 5 and Congo Red	900 °C	2 h	NA	80.46%	NA	[24]
Polypyrrole-Polyethyleneimine	Methylene Blue	65 °C	24 h	NA	99%	NA	[29]
Mixture of bentonite and charred dolomite	Basic Violet 16, Reactive Red 195	800 °C	20 min	NA	>50%	NA	[30]
Activated dolomite	Orange I	900 °C	2 h	NA	18.40%	NA	[31]
Activated sludge plants	Colour from Treated Palm Oil Mill Effluents	700 °C	1 h	NA	90–95%	NA	[33]

3.10. Limitations, Challenges and Future Strategy of Dolomite Decolourisation

- High Energy Consumption

The utilising of raw dolomite as an adsorbent is possible. However, consideration of the standard reaction and natural weathering process that occurs over thousands or even millions of years to achieve the objective is a huge consideration. To enhance the reaction process, pretreatment must be performed on the dolomite, such as thermal activation. Thermal activation removes chemically bound water, which may increase the porosity, resulting in surface area effects and disordering of the mineral lattice [34]. Accurate control of the thermal activation process is necessary to prevent overheating, which induces the recrystallisation of minerals and dramatically lowers the carbonation rate [35]. Due to this concern, a vast amount of utilised energy might be necessary for the pretreatment to continuously produce activated dolomite.

- Cost Efficiency

Although we have been using activated carbon for a long time, activating dolomite in water treatment is a novel technological technique. The construction cost for each unit process is presented as a function that was found to be the most practical and adaptable under many circumstances, such as those that can alter due to the designer's preference or regulatory agency regulations [36]. The most significant components of material and labour determining the cost of water treatment are the upkeep for applying activated dolomite to the system and its associated cost components [37]. Higher carbonisation temperatures produce activated dolomite with superior properties than activated dolomite generated at lower temperatures. However, these higher temperatures require significant energy, which has a high indirect cost [38].

- Sustainable Resources

In the limestone caverns of Perlis and Perak, Malaysia, dolomite is prevalent. Nevertheless, being a renewable resource, dolomite must initially be formed by replacing the calcite ions with magnesium ions, since naturally occurring dolomite requires a long time to develop [13]. Numerous natural limestone caverns must be explored and mined to maintain the dolomite supply, disrupting the surrounding environment. When using activated dolomite in water treatment, a thorough analysis of the toxicity level and potential harm to human health from ingesting treated water must be considered.

- Treatment Stability

Carbonation enables the valorisation of underused raw dolomite, reducing manufacturing and transportation costs and pollution issues whenever readily available materials

are considered [38]. Activating dolomite provides calcium (Ca^{2+}) and magnesium (Mg^{2+}), resulting in higher base saturation. At the same time, aluminium (Al^{3+}) ions are replaced by Ca and Mg, and are neutralised by OH^- ions. Consequently, the dolomite application increases the pH of the water [10]. The increment of pH is relatively high, exceeds the acceptable range and requires further treatment to reduce the pH. The adsorption of dolomite is surface-dependent, as the contaminants will bind on the available sites. At some point, the adsorption reaches equilibrium as the active sites are saturated by contaminants [39]. A sustainable method will help to sustain the treatment in the future.

- Waste Management

Once the adsorbent has become saturated with the contaminant, it can be considered a waste, generating a new environmental problem if it is not adequately disposed of [18]. Toxins may be released into the environment by the waste-saturated activated dolomite leachate. Additionally, considering dolomite is an inorganic mineral, it cannot further disintegrate unlike other biological components utilised in the filtration system [39]. Since environmental regulations for handling waste from dolomite materials are new, it may be difficult for some industries and companies to comply.

Dolomite that has been activated has the potential to acquire various beneficial properties for use in varying sorts of wastewater treatment. These characteristics enhance the water treatment process and water quality while supplying our water with essential minerals. They benefit our bodies since they contain the necessary minerals for good health. Mineral deficiencies increase an individual's likelihood of developing conditions such as osteoporosis, goitre, and migraines. Therefore, high-quality treated water is required for environmental sustainability and human health mortality.

Dolomite is an example of an inorganic substance that is difficult to break down and cannot be used by other organisms. However, it can be employed for other things, such as improving concrete or amending soil. Dolomite's crystalline structure enables us to reuse spent dolomite in water treatment at a reasonable cost to consumers. Today's industries seek more reliable, stable and cost-effectively produced technologies. Due to environmental concerns, renewable resources are one of the trendsetters in technological advancement.

4. Discussion

Dolomite is a part of the anisotropic rhombohedral crystalline system, which has a three-fold inversion axis. During the carbonisation of dolomite, rigid, porous calcium carbonate and finely ground magnesium oxide are produced [30]. Before and after carbonisation, the dolomite microstructure changes according to the temperature employed. The surface pores reveal ascending patterns. Due to ionic interactions between elements, heat treatment causes water to be released, causing the dolomite to become more porous and change in chemical composition. After the treatment, the saturated pores in the FESEM and the activated dolomite were visible in the DM and retained the dye colour [40]. The interactions between dye molecules and their positive net surface charge, which may result in ion pairs influencing the formation of a coating or layer, caused these phenomena [27].

Dolomite undergoes thermal treatment at $900\text{ }^\circ\text{C}$, altering the physical surface-based SEM examination. The surface area appropriate for dye adsorption expands because the increased temperature increases the sample's capacity to generate more pores. Dolomite activation also makes carbonation possible, allowing compounds such as CaO and MgO to proliferate on its surface and release ions during the procedure. Dolomite changes through various mechanisms, including hydrophobic partitioning, surface complexation, hydrogen bonding and coulombic interactions, and may react with the COD component. During treatment, some interactions result in heat produced as exothermic energy [21].

Figures 8 and 9 show that the adsorbent adsorption capability depends on its size. Although the adsorption rate of dolomite in the $600\text{ }\mu\text{m}$ and $1000\text{ }\mu\text{m}$ sizes may be slower during treatment, the adsorption capacity is greater than that of powdered activated dolomite. Due to the extensive pore structure on the surfaces, thermal activation improves the structure of dolomite, particularly the gravel form. Dolomite pores are classified into

various forms, including macropores, mesopores and micropores. These pores assisted in capturing, absorbing and binding to the dye and organic molecules from the dye effluent [41]. The disturbance of the dolomite physical surface enhanced the density of the mesopores and micropores.

Figures 10 and 11 show that the optimal number of adsorbents to use in the treatment is between one and three. The concentration of binding sites on the surface affected the decolourisation and reduction in COD. The ability for reduction increased as the surface area increased. However, the reduction reached an equilibrium phase after a predetermined interval and slowed down due to concentrated binding sites with the constituents in the effluent [42]. However, amongst the variables applied in the treatments, activation temperature in preparing the adsorbents affected decolourisation and COD reduction most strongly in this study.

Microbes can inhabit textile wastewater for a long time due to its contaminants, such as natural fibres, cellulose and other organic compounds that were degraded and support microbial growth. Moreover, the conditions of the wastewater favour bacterial growth in terms of pH and temperature. Various studies have been conducted to determine how to remove and control pathogens and microbes from textile wastewater. It has been reported that using oxide minerals effectively eliminates pathogens due to their alkalinity, and it was theorised that dolomite might have the same properties as other oxide minerals.

Due to its alkalinity in the pH 9–12 range, similar to limestone, activated dolomite demonstrated antibacterial properties in the current study. The fundamental mechanism of the treatment's antibacterial activities is attributed to the high concentration of hydroxyl ions produced. The highly oxidising free radicals known as hydroxyl ions exhibit tremendous reactivity with various biomolecules, which can be lethal to bacterial cells and harm their membranes, DNA and proteins [43].

5. Conclusions

Using activated dolomite as a low-economical adsorbent to clear away contaminants from textile dye wastewater contributes to creating “greener” adsorbents for a sustainable environment. With dolomite heated to 300 °C, 700 °C and 900 °C, the adsorption of real industrial and synthetic textile dye wastewater under various conditions was studied. The calcination process at high temperatures, which produced an abundance of pores on the surface, could be used to prepare the porous dolomite. The condition with the highest decolourisation rate achieved the best results across all conditions tested, with COD reductions of 98.7% and 66.7% for RTDW and 69.9% and 73.95% for STDW, respectively.

This experiment demonstrated that the treatment, as related to the number of adsorption sites and surface volume, is significantly impacted by dolomite's activation temperature, sizes, and concentration. Another advantage of this study is dolomite's demonstrated capacity to suppress microbes. This study's findings demonstrated the preliminary potential of activated dolomite, and more studies are required to completely adapt existing wastewater treatment systems to pretreating textile dye wastewater.

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Data Availability Statement: Data is available upon request.

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Conflicts of Interest: The authors declare no conflict of interest.

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