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Ammoniacal nitrogen, chemical oxygen demand, and color reduction in rubber processing industry effluent using zeolite

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ABSTRACT

The effluent from the rubber processing industry usually contains a high concentration of organic compounds, suspended solids, color, nitrogen, and other pollutants. If an excessive amount of ammonia nitrogen is discharged into bodies of water, it may cause eutrophication and the death of various aquatic creatures. The goal of this experimental design was to investigate the effectiveness of zeolite for ammoniacal nitrogen, chemical oxygen demand (COD), and color removal from rubber processing industry effluent. The samples of rubber processing industry effluent for this study were acquired directly from the discharging point of a manufacturer in Kluang, Malaysia. The effects of optimal adsorbent dose, shaken speed, contact time, and pH were determined for ammoniacal nitrogen, COD and color removal. The obtained result reveals that the best batch adsorption experiment of ammoniacal nitrogen, COD, and color removal was attained at pH 7, ZEO dosage 4.0 g, contact time 120 min, and shaken speed 200 rpm respectively. The best efficiency removal of ammoniacal nitrogen, COD, and color was achieved based on dosage generally in the range of 75% for ammoniacal nitrogen, 69% for COD, and 79% for the color of all batch experiments conducted. The kinetic equilibrium model data fitted well with the pseudo-first and second model. The pseudo-second-order better fitted the equilibrium data over the entire concentration range studied. The pseudo-second-order described the best coefficient of determination compared to the pseudo-first-order. The coefficient of determination for pseudo-second-order was generally in the range of 0.9958 for ammoniacal nitrogen, 0.9978 for COD, and 0.9984 for the color of all kinetics studies conducted.

Keywords: Adsorption kinetics model; Ammoniacal nitrogen; Chemical oxygen demand and color; Rubber effluent; Zeolite

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1. Introduction

Natural processing rubber is one of the most valuable and important agricultural products in South East Asian countries. Conversely, the natural rubber processing industry has caused numerous impacts on the environment including air, water bodies, and odor pollution [1,2]. A large quantity of wastewater was generated from the processing of raw natural rubber as its operation required a huge amount of water [3]. This effluent contains a higher concentration of ammoniacal nitrogen, organic constituent, biological oxygen demand (BOD), nitrate, phosphorus, and total suspended solid [1,2]. The direct discharge of this effluent into water bodies like streams, drains, and rivers result to affect the ecosystem of aquatic life and also may cause eutrophication [4,5]. Several systems have been developed such as anaerobic-cum-facultative lagoon system, anaerobic-cum-aerated lagoon system, aerated lagoon system, and oxidation ditch system have been developed for the treatment of rubber industry wastewater [5]. Despite their low cost, these systems required a longer period of effluent treatment, a considerably larger area, odor problems, and also high operating and maintenance costs [3,5].

The adsorption technique has shown effective and widely used technique for the removal of contaminants from an aqueous solution [6]. Activated carbon (AC) is most commonly and widely used as an adsorbent for the treatment of wastewater because it has a high surface area capability to adsorbed organic matter but due to its high price its usage is limited [7]. Furthermore, activated carbon (AC) is remain consider quite an expensive adsorbent, higher the quality, higher the price [8,9]. Thus, a cost-effective material has led to a search. Over the past 20 y, many researchers have examined the feasibility of cost-effectiveness, commercially available materials which are easier to reproduce and reuse as often as possible [10].

The utilization of zeolite-based adsorbent has a specific advantage over conventional techniques that were applicable for the treatment of industrial effluent. Natural zeolite is a kind of porous substance with a higher surface area. It possesses a negative charge, which permits it to adsorbed cations [11]. Zeolite possesses high cation-exchange capacity (CEC) and hence have great potential for application in the ammoniacal nitrogen removal from wastewater [12]. The removal of ammoniacal nitrogen by utilizing natural zeolite adsorbent is regarded as an effective and competitive treatment technique because of its cost-effectiveness and relatively easy to use and operation [13]. Natural zeolite (clinoptilolite) has a three-dimensional crystalline structure and its typical cell formula is given as $Na_6[(A1O_2)_6(SiO_2)_{30}]·24H_2O$ [14]. They contain exchangeable alkali and alkaline earth metal cations such as K^+, Na^+, Ca^{2+}, and Mg^{2+} that maintain a neutral charge. The microporous crystalline structures of zeolite allow ionic species exchange with a diameter that fitted within the entry port of inner zeolite frameworks, while large species are excluded, resulting in ions sieving capabilities that can be used in a range of commercial applications [15].

The author in this study utilized ZnO nanoparticles for photocatalytic degradation of phenol, and the effects of initial concentration, Dosage, contact time, and pH parameters were investigated. The optimal process condition in nano-photocatalytic UV/ZnO were obtained in 10 mg L^{-1}, contact time of 30 min at pH 5. The finding result revealed that the nano-photocatalytic technique can effectively remove phenol from an aqueous solution [26]. A chemical co-precipitation approach was employed to produce magnetic zeolite nanocomposite with an average diameter of 90–100 nm, which was then used to adsorb dimethyl phthalate (DMP) from an aqueous solution. The findings revealed that increasing the contact time and dosage effectively increases the adsorption performance efficiency [27,28].

MGO nanoparticles (MGO-NPs) were synthesized and their characteristics were characterized using EDX, XRD, SEM, VSM, FTIR, TEM, and N_2 adsorption/desorption techniques. Determine the mutual and individual effect of variables on the removal process, such as pH, sonication period, DEP concentration, and adsorbent dosage. The Langmuir fits best with equilibrium data and adsorption kinetic follows the pseudo-second-order [29]. The author of this study utilized hybrid ultrasound to purify $GOFe_3O_4$ from DNP solution their characteristics were characterized by BET, XRD, VSM, FTIR, SEM, TEM, and various parameters on the removal of DNP were estimated and optimized using RSM. The Langmuir fits best with equilibrium data and adsorption kinetic follows the pseudo-second-order [30].

The goal of this research work is to optimize and model the degradation of dimethyl phthalate (DMP) from an aqueous solution utilizing $UVC/Na_2S_2O_8/Fe^{2+}$ according to the RSM approach. The higher efficiency removal 97% and TOC removal 64.2% were achieved under the optimal condition such as in DMP 10 mg/L concentration, contact time 90 min at pH 11, Fe^{2+} = 0.075 mM/L and SPS concentration = 0.601 mM/L. The finding result revealed that the adsorption technique can effectively remove lead using iron oxide nanoparticles and carbon (ION/C) composite [31,32].

The author in this study evaluates Taguchi and CCD technique in the adsorption process. The parameter chosen to be contact time, pH, and initial concentration, and removal of lead (Pb) were chosen for designated responses. The findings revealed that in an optimal condition, the efficiency removal of Pb was 80%. The R-square value of both Taguchi and CCD techniques was >0.95, which indicates that both methods were appropriate and compatible with each other [33]. The goal of this study was to investigate fluoride adsorption isotherms and kinetics using a magnetic chitosan/graphene oxide (MCGO) composite. The optimal fluoride was obtained at pH 5, contact time 136 min at pH 5 and the efficiency removal was achieved at 91% in optimum conditions [34].

The author of this research was to compare Greek bentonite and zeolite Metaxades, forms toward adsorption from aqueous solutions. Organo-forms were prepared via a chemical modification process including hexadecyltrimethylammonium bromide (HDTMABr). According to the findings, HDTMA is potentially utilized as a cost-effectiveness adsorbent for the removal of U(VI) in wastewater [39,40].

The author of this research utilized biochar for oxidized reduction and reducing nitrogen from aqueous solutions. The Langmuir fits best with equilibrium data and adsorption kinetic follows the pseudo-second-order [41].

The purpose of this present research study is to determine the effectiveness of zeolite as an adsorbent and the use of zeolite is considered a novelty for the removal of ammoniacal nitrogen, chemical oxygen demand (COD), and color on rubber effluent treatment. The best optimum dosage, pH, shaking speed, and contact time was also investigated. The optimization of these factors may improve the process competency.

2. Material and methods

2.1. Preparation of material

Natural zeolite namely (clinoptilolite) was supplied from PT Zeolita Jakarta, Indonesia. Zeolite was crushed and sieved to obtained particle size between 106 to 150 μm using a ceramic ball mill. The samples were placed in the oven and dry at 105°C for 24 h (to eliminate excess content of moisture), and then placed the sample in a desiccator to exclude atmospheric moisture until it is been tested.

2.2. Wastewater sampling

In this research study, rubber processing industry effluent samples were directly collected from the point of discharge located in Kluang, Malaysia. The obtained samples were immediately transported to the laboratory and preserved in a cold room at 4°C before minimizing further reactions. The characterization of the samples was performed and analyzed according to the Standard Methods for Examination of Water and Wastewater (APHA, 2012) [16].

2.3. Batch adsorption experiment

The batch adsorption experiment was conducted to investigate the optimum range of variables including optimum dosage, shaking speed, contact time, and pH respectively. Each variable process that needed to optimize was examined and separately monitored. The batch experiment was carried out with zeolite as the media and 100 mL of rubber industry effluent sample in a 250 mL titration flask. The top cover of the flask is wrapped with Parafilm M, laboratory film for ensuring a satisfactory agitation process. The prepared sample flask was agitation with an orbital shaker (Sartorius, Germany). The sample flask was then removed from the shaker and allowed to settle before the supernatant was collected for ammoniacal nitrogen analysis.

2.4. Rubber industry effluent

COD and ammoniacal nitrogen were determined by reactor digestion closed reflux colorimetric method (5220-D) and Nesslerization method respectively. The platinum cobalt (Pt-Co) method was used to measure color using a true color test at 455 nm using HACH/DR6000 spectrophotometer. Each sample was individually filtered before measurement through a new filter paper with a pore of 0.45 μm. All procedures and analyses were extracted from Standard Methods for the Examination of Water and Wastewater [16]. All experiments were performed in triplicate to achieve consistent results at room temperature 25°C ± 2°C [42–45].

3. Results and discussion

3.1. Characteristics of adsorbent

Table 1 shows the chemical characterization concentration of zeolite in percentage using X-ray fluorescence (XRF) analyzer. Considerably, silicon dioxide (SiO_2) and aluminum oxide (Al_2O_3) is the main zeolite compound found in higher percentages (64.5%) and (15%) respectively. The characterization experiment was performed on a poor percentage of sodium oxide (Na_2O) and magnesium oxide (MgO) was found lower percentage (0.8%) and (0.7%) and a higher percentage of calcium oxide (CaO) and potassium oxide (K_2O) were (3.23%) and (3.55) respectively [17]. Zeolite characterization results have a rough surface with open cavities and a 3D structure composed of octahedra (Al_2O_3) and/or aluminum tetrahedra (SiO_2) of the framework [18,19]. The microstructure of the zeolite adsorbent materials is presented in Fig. 1 showed a dense structure with a closely parked pore arrangement. Evidence of cracks was observed in some parts of the surface.

3.2. Comparison study of ammoniacal nitrogen, COD, and color with another adsorbent

Mainly, the usage of zeolite is mostly considered a novelty. The ZEO is analyzed and the results are compared to previous study findings. In comparison to other mixed adsorbent materials, single ZEO has shown higher performance as illustrated in Table 2. The table demonstrates that ZEO shows higher ammoniacal nitrogen, COD, and color removal than the other findings.

3.3. Chemical analysis of natural rubber wastewater

Rubber processing industry effluent samples were directly collected from the point of a discharge is located in Kluang, Johore, Malaysia. The samples were analyzed

Table 1
XRF analysis data of zeolite

Formula	Chemical characterization (%)
Original (g)	7
Added (g)	3
SiO_2	64.5
Al_2O_3	15
K_2O	3.55
CaO	3.23
Fe_2O_3	0.94
C	1
Na_2O	0.8
MgO	0.7
TiO_2	0.16
SrO	0.12

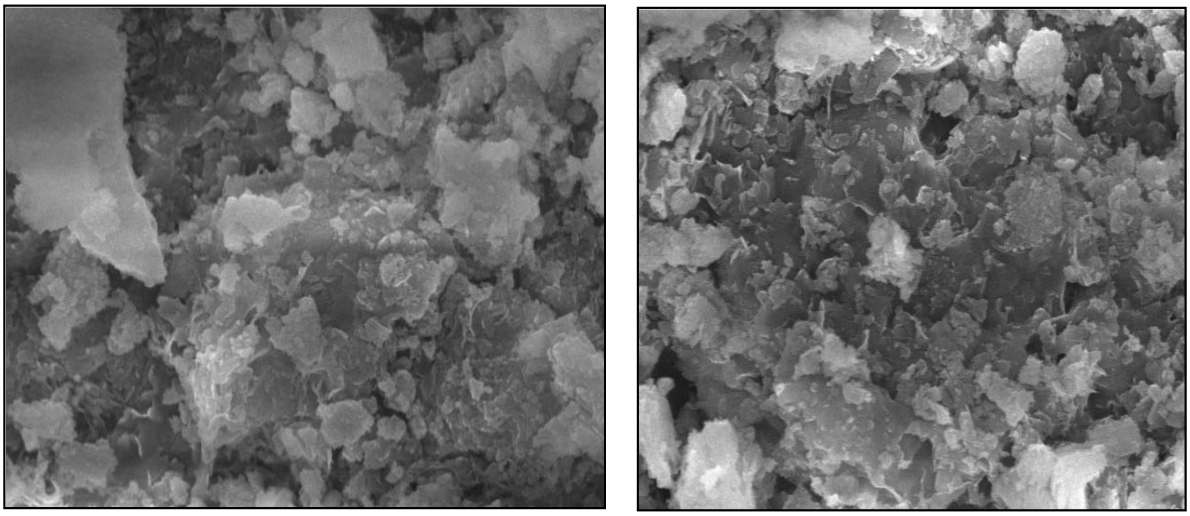

Fig. 1. Surface physical morphology of ZEO (a) before and (b) after adsorption.

Table 2
Comparison to previous study findings adsorbent

References	Type of wastewater	Absorbent	Parameter	Removal efficiency (%)
[35]	Lake wastewater	Green mussel shell	NH_4^+	31.28
			COD	44.45
[36]	Leachate	Cockle shell	COD	55
[37]	Leachate	Activated carbon and green mussel	COD	83
			NH_3–N	63
[38]	River water	Cockle shell	COD	38.8
Present study	Leachate	Zeolite	NH_3–N	75
			COD	69
			Color	79

and examined to identify the initial characterization concentration of effluent namely COD, BOD_5, pH, total suspended solids (TSS), ammoniacal nitrogen, color, turbidity, zinc, iron, and copper. The characterization of the effluent is illustrated in Table 3. The concentration value of these samples was COD, BOD_5, pH, TSS, ammoniacal nitrogen, color, turbidity, zinc, iron, copper 5,260 mg/L, 3,350 mg/L, 9.3, 500 mg/L, 55 mg/L, 345 Pt.Co, 130 NTU, 0.266 mg/L, 0.0 8 mg/L, and 0.05 mg/L, respectively. The initial characterization values are higher as compared to an acceptable condition for discharge of industrial effluent regulation 2009 [20]. It must be treated before being released into another body of surface water.

4. Adsorption studies

4.1. Effect of different optimum dosages towards ammoniacal nitrogen, COD, and color

The investigation of optimum dosage is considered an important key factor in the quantitative uptake of contaminants. The effect of different optimum dosages varies from (0.0 to 8.0 g) towards the percentage reduction of ammoniacal nitrogen, COD, and color as shown in Fig. 2. The reduction percentage efficiencies of ammoniacal nitrogen, COD, and color increase with increasing of zeolite adsorbent dosage from 0.5 to 4.0 g respectively. The best optimum result was considered at 4.0 g with removal percentage of ammoniacal nitrogen, COD and color were (75%, 69%, and 79%) respectively. Subsequently, further increments in the zeolite dosage do not increase in removal percentage of factors because utmost all ammoniacal nitrogen, COD, and color were adsorbed in rubber effluent when further increment in dose was 4.5 g. More rapidly increasing in adsorption with increasing the amount of adsorbent due to higher surface area and accessibility to more adsorption sites [21,22].

4.2. Effect of different optimum pH towards ammoniacal nitrogen, COD, and color

The pH initial concentration of rubber industry effluent is considered an important key parameter, which controls the adsorption process, mainly the adsorption capacity. The effect of different optimum pH varies from 3 to 12 towards the percentage reduction of ammoniacal

Table 3
Characteristics of natural rubber effluent

Parameters	Value	Discharge of Industrial effluent of Standards A and B	
		A	B
Biological oxygen demand (BOD), mg/L	3,350	20	50
Chemical oxygen demand (COD), mg/L	5,260	80	200
Total suspended solids, mg/L	500	50	100
Ammoniacal nitrogen, mg/L	55	10	20
Color, Pt.Co	345	100	200
Turbidity, NTU	130		
Zinc, mg/L	0.266	2.0	2.0
Iron (Fe), mg/L	0.08	1.0	5.0
Copper (Cu), mg/L	0.05	0.20	1.0
pH	9.3	6.0–9.0	5.5–9.0

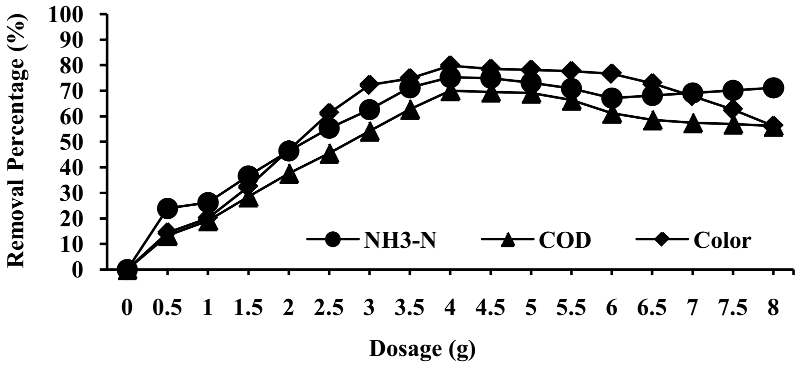

Fig. 2. Percentage of ammoniacal nitrogen, COD, and color removal against different zeolite dosage.

nitrogen, COD, and color using zeolite adsorbent as shown in Fig. 3. The reduction percentage efficiencies of ammoniacal nitrogen, COD, and color increase with increasing pH concentration from 3 to 7 respectively. The best optimum result was considered at pH 7 with the removal percentage of ammoniacal nitrogen, COD, and color (71%, 70%, and 74%) respectively. Subsequently, with further increments in the concentration of pH solution 9, the removal percentage start decreasing. This finding result correlates with other researchers [13].

4.3. *Effect of different optimum shaking speeds towards ammoniacal nitrogen, COD, and color*

The effect of stirring rate is considered an important key factor in the adsorption phenomenon since this affects the solute distribution in bulk solution and the formation rate of outer boundary film [21,22]. The effect of different optimum shaking speeds varies from (00, 50, 100, 125, 150, 175, and 200 rpm) towards the percentage reduction of ammoniacal nitrogen, COD, and color as shown in Fig. 4. The reduction percentage efficiencies of ammoniacal nitrogen, COD, and color increase with an increase in shaking speed from 50 to 150 rpm respectively. The best optimum result was considered at 150 rpm with the removal percentage of ammoniacal nitrogen, COD, and color (55%, 81%, and 81%) respectively. Subsequently, further increments in the shaking speed do not increase in removal percentage of parameters because utmost ammoniacal nitrogen, COD, and color were adsorbed in rubber effluent when further increment in speed to 200 rpm. More rapidly increasing in adsorption with increasing the rate of speed reducing surrounding particles boundary layers, hence increasing external film transfer coefficient, and hence the percentage ammonia nitrogen removal [23].

4.4. *Effect of different optimum contact times towards ammoniacal nitrogen, COD, and color*

The investigation of optimum contact time is considered an important key parameter in the quantitative uptake of contaminants. The effect of different optimum contact times varies from 5 to 300 min towards the percentage reduction of ammoniacal nitrogen, COD, and color as shown in Fig. 5. The reduction percentage efficiencies of ammoniacal nitrogen, COD, and color increases with increasing time from 5 to 120 min respectively. The best optimum result was considered at 120 min with the removal percentage of ammoniacal nitrogen, COD, and color being (75%, 80%, and 84%) respectively. Subsequently, further increments in the contact

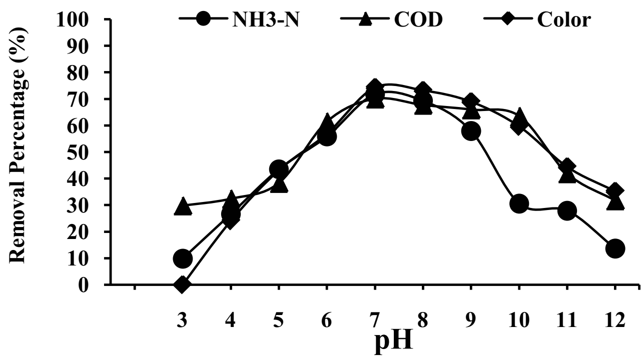

Fig. 3. Percentage of ammoniacal nitrogen, COD, and color removal against different pH.

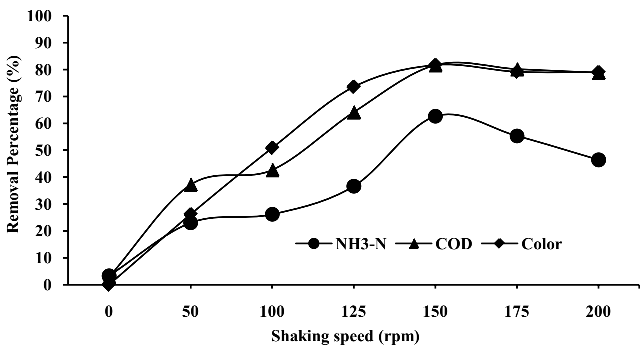

Fig. 4. Percentage of ammoniacal nitrogen, COD, and color removal against different shaking speed.

time do not increase in removal percentage of parameters because utmost ammoniacal nitrogen, COD, and color were adsorbed in rubber effluent when further increment in dose was 150 min.

5. Kinetic studies

Adsorption kinetics models were subjected to examine the experiment data to investigate the rate of adsorption and probable adsorption mechanism onto the zeolite. In this research study, the kinetics model was applying pseudo-first-order and pseudo-second-order.

Pseudo-first-order kinetics model is considered to measure adsorption uptakes with respect to time and was directly proportional to the difference between saturation concentration and the amount adsorbed [23]. The pseudo-first-order kinetic model expression is stated below [24,25].

$$\ln\left(q_e - q_t\right) = \ln q_e - \left(\frac{K_1 t}{2.303}\right) \quad (1)$$

where "k_1" indicates the pseudo-first-order constant adsorption rate, "q_t and q_e" indicate values of the adsorbed mass per unit mass at equilibrium, and "t" denotes time respectively.

The experimental data of pseudo-first-order were not best fitted as it can be seen from the lower correlation coefficient (R^2) value of 0.7272. The pseudo-second-order kinetics model assumption is based on chemical sorption as a rate-limiting step and is known as [2].

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

where "k_2" denotes the pseudo-second-order constant equilibrium adsorption rate (g/mg min).

The obtained result indicates that the experimental data fitted better to pseudo-second-order for adsorption of ammoniacal nitrogen, COD, and color from rubber industry effluent. The value of the coefficient of determination (R^2) is approximately near 1. The highest values of coefficient of determination (R^2) indicate that the parameters of adsorption kinetics are well described by the model [9].

The adsorption maximum capacities (q_e) determine using the pseudo-second-order expression were close to experimental values.

The result obtained indicates that the pseudo-second-order kinetics model provided the best coefficient of determination (R^2) for ammoniacal nitrogen, COD, and color onto the zeolite (Figs. 6–11).

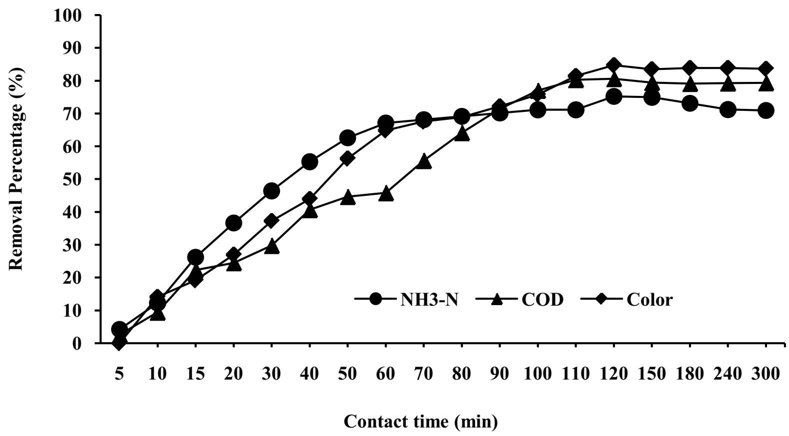

Fig. 5. Percentage of ammoniacal nitrogen, COD, and color removal against different contact time.

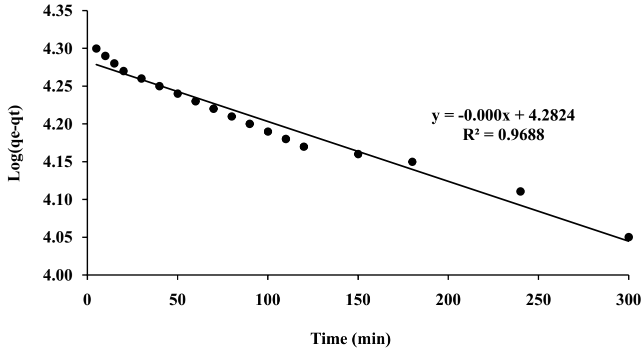

Fig. 6. Pseudo-first-order kinetic model for NH_3–N.

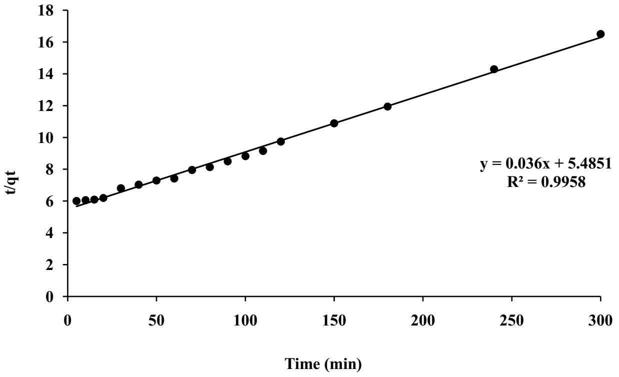

Fig. 7. Pseudo-second-order kinetic model for NH_3–N.

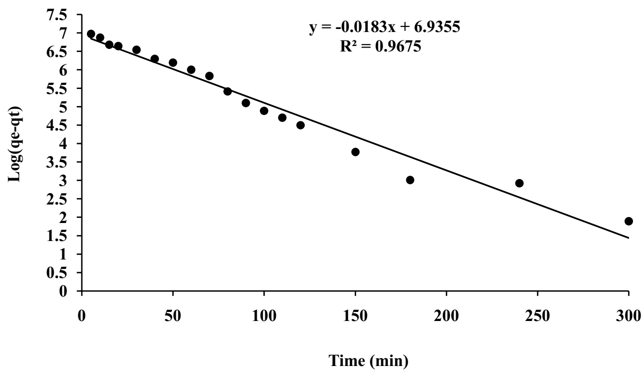

Fig. 8. Pseudo-first-order kinetic model for COD.

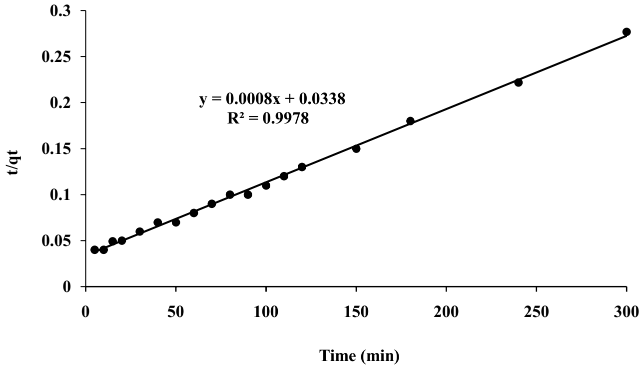

Fig. 9. Pseudo-second-order kinetic model for COD.

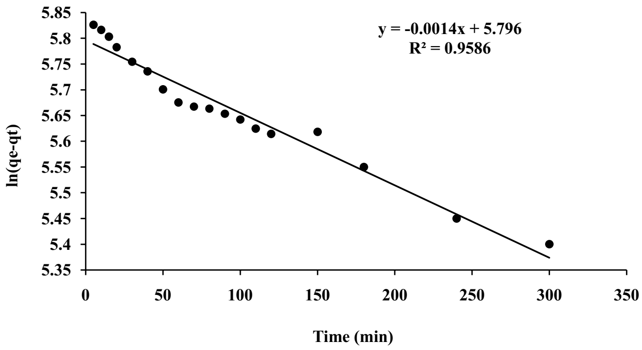

Fig. 10. Pseudo-first-order kinetic model for color.

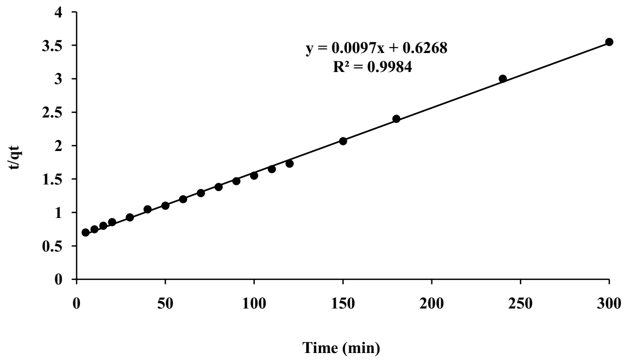

Fig. 11. Pseudo-second-order kinetic model for color.

6. Conclusion

This research study shows that the utilization of zeolite as an adsorbent is cost-effectiveness and environmentally friendly acceptable material and it can remove pollutants of over 80% namely ammoniacal nitrogen, COD, and color from the rubber industry effluent. The best batch adsorption experiment optimum dosage result was considered at 4.0 g with removal percentage of ammoniacal nitrogen, COD and color were (75%, 69%, and 79%) respectively. The best optimum result was considered at pH 7 with removal percentage of ammoniacal nitrogen, COD and color were (71%, 70%, and 74%) respectively. The best optimum result was considered at 150 rpm with removal percentage of ammoniacal nitrogen, COD and color were (55%, 81%, and 81%) respectively. The best optimum result was considered at 120 min with the removal percentage of ammoniacal nitrogen, COD, and color being (75%, 80%, and 84%) respectively. The kinetic equilibrium model data were fitted well to the pseudo-first-order and pseudo-second-order kinetics model. The pseudo-second-order better fitted the equilibrium data over the entire concentration range studied. The pseudo-second-order kinetic model described the best coefficient of determination compared to the pseudo-first-order kinetics model. The coefficient of determination for pseudo-second-order was generally in the range of 0.9958 for ammoniacal nitrogen, 0.9978 for COD, and 0.9984 for the color of all kinetics studies conducted. However, more research work should be conducted to determine the effectiveness of zeolite for removing other pollutants from rubber industry effluent namely total suspended solids, zinc, iron (Fe), copper (Cu), and many others.

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