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ИССЛЕДОВАНИЕ АДСОРБЦИИ КРАСИТЕЛЕЙ ИЗ ВОДНОГО РАСТВОРА С ИСПОЛЬЗОВАНИЕМ КРАСНОГО ШЛАМА, АКТИВИРУЕМОГО СЕРНОЙ КИСЛОТОЙ

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Сточные воды текстильной промышленности содержат большое количество различных красителей, которые являются довольно токсичными и должны быть отделены перед выбросом в окружающую среду. Они часто обладают высокой устойчивостью к биодеградации, и поэтому их трудно перерабатывать. Применение адсорбентов природного происхождения, особенно промышленных отходов, является одним из наиболее привлекательных решений для обработки сточных вод из-за его высоких социально-экономических преимуществ. В этом исследовании была изучена адсорбционная способность некоторых обычных красителей, таких как Red 195 и DirectYellow 132 на красном шламе, активированном серной кислотой. В процессе кислотной активации часть оксида алюминия, оксид железа на красном шламе будет растворяться, тем самым увеличивая удельную площадь поверхности оставшейся твердой фазы (с 55 м^2/г до 92 м^2/г), количество растворенного красного шлама составляет около 30% вес. Твердый остаток используется в этом исследовании адсорбции. Раствор, полученный после активации, включает соли сульфата железа, сульфата алюминия, используемые в качестве коагулянта для очистки сточных вод. Результаты показали, что для обоих красителей pH 5 наиболее подходит для процессов адсорбции. Кинетика адсорбции была описана кинетическим уравнением псевдо-второго порядка. Константы скорости модели второго порядка для адсорбции DY132, RR195 на RMA в растворе с концентрацией 100 мг·л$^{-1}$, pH =5 составляют 1,48 и 1,95·10^{-2} g/(mg·min) соответственно и равновесные адсорбционные емкости составляют 42,74 и 54,95 мг·г$^{-1}$ соответственно. Данные адсорбции были хорошо сопоставлены с моделью изотермы Ленгмюра, максимальная адсорбционная способность была определена 48,54 и 84,31 (мг/г) для Red 195 и DirectYellow соответственно.

Ключевые слова: красный шлак, активируемый кислотой красный шлак, адсорбционные сборы, бюджетный адсорбент

STUDY ON DYES ADSORPTION FROM AQUEOUS SOLUTION USING RED MUD ACTIVATED BY SULFURIC ACID

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The textile industry wastewater contains the majority of different dyes which are quite toxic and should be removed before disposal. They are often highly resistant for biodegradation and hence are difficult to be treated. The application of adsorbents of natural origin, especially industrial waste, is one of the most attractive solutions for wastewater treatments due to its high socio-economic advantages. In this study, the adsorption capacity of acid activiated red mud for some conventional dyes such as Reactive Red 195 and Direct Yellow 132 was investigated. In this acid activation process part of aluminum oxide, iron oxide on red mud will be dissolved into solution, thereby increasing the specific surface area of the remaining solid phase (from 55 m^2/g to 92 m^2/g). The amount of red mud dissolved in the solution is about 30% weight. Solid residue is used in this adsorption study. The solution obtained after activation which includes iron sulfate salts, aluminum sulfate used as a coagulant for wastewater treatment. The results showed that, for both dyes, pH 5 is most suitable for the adsorption processes. The adsorption kinetic was based on the pseudo second-order kinetic equation. The rate constants of the second-order model for adsorption of DY132, RR195 on RMA in a solution with a concentration of 100 mg·l^{-1}, pH = 5 are 1.48 and 1.95·10^{-2} g/(mg·min), respectively, and the equilibrium adsorption capacities are 42.74 and 54.95 mg·g^{-1}, respectively. The adsorption data were well matched to Langmuir isotherm model. The maximum adsorption capacities were found to be 48.54 and 84.31 (mg·g^{-1}) for Reactive Red 195 and Direct Yellow 132, respectively.

Key words: red mud, acid activated red mud, dyes adsorption, low-cost adsorbent

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INTRODUCTION

Dye stuff is an organic pollutant discharged from textile, printing, paper, and leather industries. Discharge of dyes causes environmental problems. Dyes reduce the penetration of light and photosynthesis, destroy the ecosystem. In addition, some dyes are either toxic, or mutagenic and carcinogenic. There are many methods that have been studied to treat dyes such as flocculation [1, 2], chemical oxidation [3, 4], electrochemical [5, 6], Fenton [7-9], filtration [10, 11], biodegradation [12, 13], adsorption [14] etc. In this, adsorption is considered effective and most economical.

Red mud is a solid waste generated during the Bayer process of alumina production in the aluminum industry. It is reported that it removes several significant dye pollutants, such as congo red, acid violet, methylene blue, rhodamine B,.... [15-17].

In order to improve the adorption capacity of red mud, various activation methods have been investigated. Gupta et al. treated red mud by H_2O_2 at room temperature for 24 h to oxidize adhering organic matter and washed repeatedly with doubly distilled water [18].

Ratnamala et al. reported on the utilization of the acid treated red mud as an adsorbent for removal of

Remazol Brilliant Blue dye, a reactive dye from dye synthetic water [19].

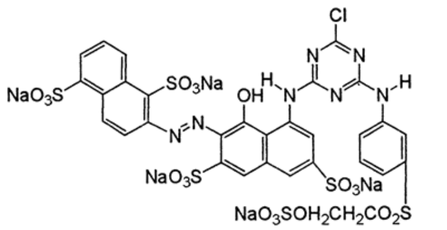
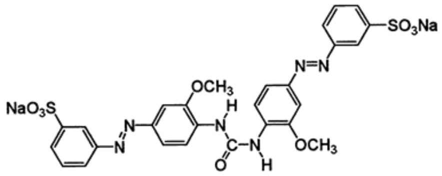
In this paper, following the previous studies [20] we investigated the adsorption capacity of some commercial dyes, Direct Yellow 132 and Reactive Red 195, on acid activated red mud.

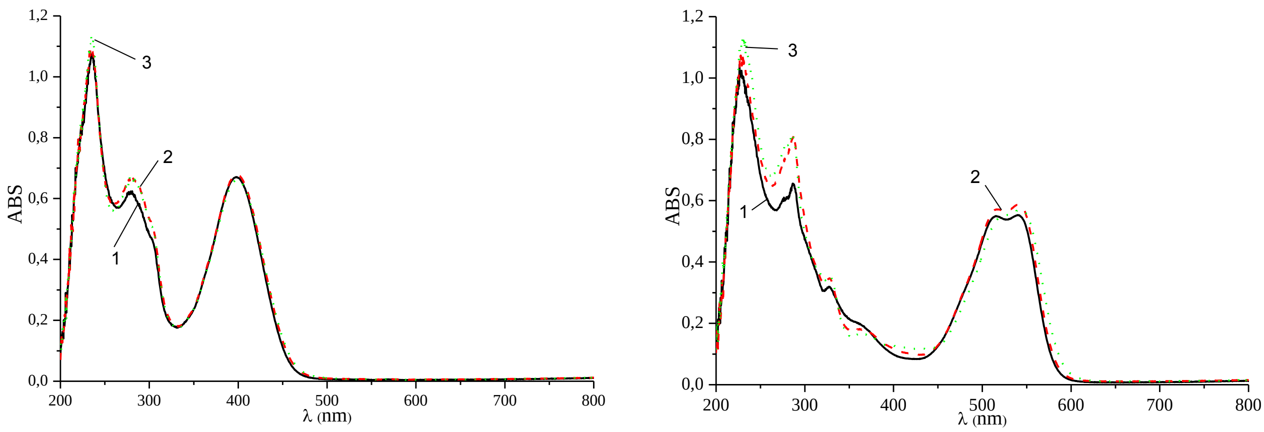
EPERIMENTAL PART

In this study, red mud was obtained from Alumin Tan Rai plant, Lam Dong, Vietnam. Red mud was activated by 2M H_2SO_4 with liquid/solid ratio 2 mL/g, stirred and heated at 90 °C within 2 h according to our recent work [20-23]. In this acid activation process part of aluminum oxide, iron oxide on red mud will be dissolved into solution, thereby increasing the specific surface area of the remaining solid phase (from 55 m^2/g to 92 m^2/g), the amount of red mud dissolved in the solution is about 30% weight [21]. Solid residue is used in this adsorption study. The solution obtained after activation which includes iron sulfate salts, aluminum sulfate used as a coagulant for wastewater treatment [21, 24]. The dyes used in adsorption experiment are commercial dyes Reactive Red 195 (RR195) and Direct Yellow 132 (DY132) from Ningbo Mingzhou Chemical DyestuffCo., China. The concentration of dyes in the solution before and after adsorption was determined by photometric analysis method. The maximum absorption wavelength of dyes according to pH media was determined by Ultraviolet-Visible Spectroscopy on the CINTRA 40, GBC spectrophotometer (America) (Fig. 1). Construction of calibration curve for determination of dyes concentration (corresponding to different pH environments) by measuring ABS absorption at λ_{max} corresponding to HACH DR/2010 spectrophotometer in the Institute for Tropical Technology – Vietnam Academy of Science and Technology (Table 2).

Table 1

Properties of dyes under study
***Таблица 1*. Характеристика используемых красителей**

Name	Molecular formula	Molecular structure	Wavelength (nm)
Reactive Red 195 (RR195)	$C_{31}H_{19}ClN_7Na_5O_{19}S_6$ M_W =1136.32(g/mol)		540
Direct Yellow 132 (DY132)	$C_{27}H_{22}N_6Na_2O_9S_2$ M_W = 684.61(g/mol)		400



a b

Fig. 1. UV-Vis defraction of DY132 (a) and RR195 (b)
Рис. 1. UV-Vis дифракция DY132 (a) и RR195 (b)

Table 2
The calibration curve of the concentration of DY132 and RR195
***Таблица 2.* Калибровочная кривая концентрации DY132 и RR195**

pH	DY132		RR195	
	Calibration curve equation	R^2	Calibration curve equation	R^2
2	y = 0.031x + 0.0018	0.9983	y = 0.026x + 0.0043	0.9943
6	y = 0.031x – 0.0004	0.9988	y = 0.026x + 0.0024	0.9987
10	y = 0.031x + 0.0018	0.9982	y = 0.024x + 0.0042	0.9991

Adsorption of dyes on the H_2SO_4 – activated red mud was examined by a bath method at room temperature (25 °C); the factors influencing to adsorbent capacity were investigated: pH, contact time and initial concentration of the dyes solution.

- *Effects of pH:* the concentration of initial dyes C_0 = 30 mg/l, the amount of adsorbent is 1 g/l, the adsorption time is 120 min, the pH varies from 2 to 10. The pH of the dye solutions was adjusted to the desired values with 0.1M NaOH or 0.1M HCl solutions. Analyze the dyes concentrations after adsorption and calculate the adsorption capacity, thereby determining the optimum pH.

- *Adsorption kinetics studies:* put in 250 mL of dye solutions with concentration 30 mg/L, 70 mg/L and 100 mg/L to the triangular flask, pH and the amount of adsorbent determined from the above experiments. After a certain time interval from 1 min to 120 min, determine the remaining dye concentration in the solution and calculate the adsorption capacity.

- *Asorption isotherm studies:* Prepare triangular flask containing 50 mL of a dyes with a concentration of 30 to 300 mg/L; the amount of adsorbent is 1 g/L; adjust pH to optimal value. Determine the concentration of dyes at equilibrium state and calculate adsorption efficiency and adsorption capacity. From the adsorption results, the isotherms were analyzed by the two commonly used Langmuir and Freundlich models [15-20] have been studied.

RESULTS AND DISCUSSION

1. Effect of activation methods on the adsorption capacity of red mud

After activating the red mud, determine the adsorption capacity of the activated red solution (RMA) dyes for comparison with the raw red solution (RM). The bath adsorption tests were carried out in a container with 50 ml of solution DY132 and RR195, an initial concentration of 30 mg/l, the amount of adsorbent was 1 g/l, the contact time was 120 min, the adsorption capacity q was calculated and represented in Fig. 2.

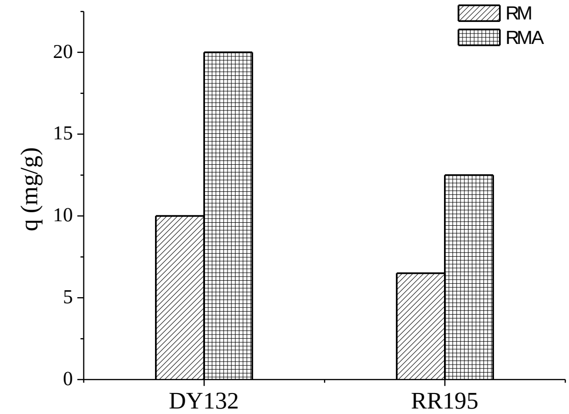


Fig. 2. DY132, RR195 adsorption capacity of RM, RMA
Рис. 2. DY132, RR195 адсорбционная способность RM, RMA

Dyes are all in the form of anions in aqueous solution. In all cases, red mud, which is activated by acid, increases the adsorption capacity. Activated by acid red mud, in addition to increasing its own specific surface area (55 to 92 m^2/g [20-23]) also protonates the active center on the surface.

2. Effect of pH

The pH of the solution is an important variable that controls the adsorption of metal ions on the surface of the adsorbent. The adsorption capacity q was determined and presented in Fig. 3.

It can be seen that the process of dyes adsorption is beneficial in acidic media, where the pH> 5 adsorptive capacity of all dyes is greatly reduced. This can be interpreted similarly to the adsorption of other anions on red mud, dye anions, which tend to form electrostatic bonds with positive charge centers on the adsorbent surface in low pH environments [22, 23-25, 26]. In the case of a high pH, a negatively charged surface of the material, adsorption will not occur. Therefore, pH = 5 was chosen for further experiments.

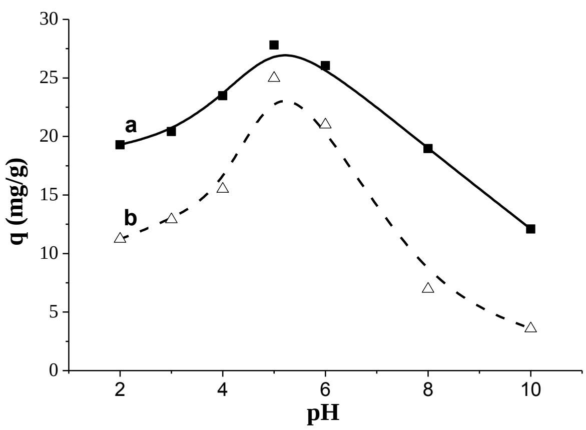


Fig. 3. Effects of pH on the adsorption capacity: (a) DY132; (b) RR195 on RMA
Рис. 3. Влияние pH на адсорбционную способность: (a) DY132; (b) RR195 на RMA

3. Kinetics studies of the dyes adsorption on RMA

The adsorption efficiency H of dyes were investigated with time of adsorption in the solution 30 mg/l, 70 mg/l and 100 mg/l dyes at pH 5, the amount of adsorbent RMA was 1 g/L. The results are presented in Fig. 4.

At a dye concentration of ≤ 70 mg /l, the adsorption rate was very high, so the data were not used at this concentration to analyze the kinetics of adsorption. Experimental data on the adsorption of dyes in a solution of 100 mg/l for a period of less than 60 min were analyzed by pseudo-first-order and second-order equations, the results were presented in the Table 3. Results showed that the correlation coefficient for the second order model is very high and the calculated values q_e are close to the experimental values q_t, that better fits the experimental data. The dye adsorption rate was calculated by the formula: $v_0 = k_2 \cdot q_e^2$ and the time ($t_{1/2}$) is the moment when the adsorption capacity reaches 50% qe (meaning 0.5 of the equilibrium value qe) and the time ($t_{0,99}$) is close to equilibrium then the adsorption capacity reached 99% qe, were determined and the results were presented in Table 4. Those showed that the adsorption of DY132 was faster than RR195.

Compared with DY132, RR195 has a significantly higher molecular weight (M_{RR195} = 1136,32 g/mol) and much more bulky structure, so that the rate of adsorption is significantly lower than that for DY132, the time until equilibrium is also longer. However, in all cases the the $t_{1/2}$value is quite small, less than 3 min.

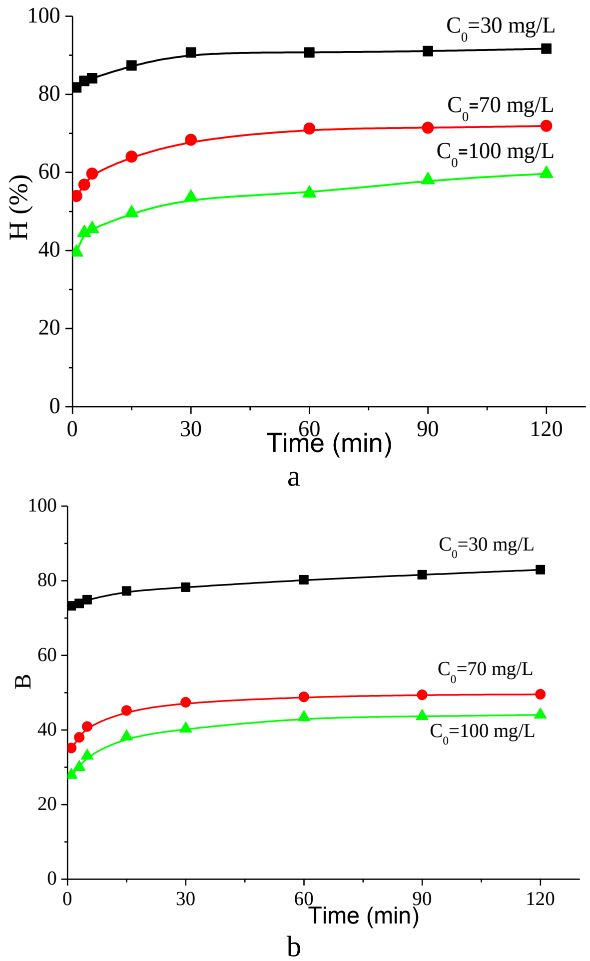


Fig. 4. Adsorption efficiency (a) DY132; (b) RR195 of RMA over time of contact, with C_0 of 30, 70 and 100 mg/l
Рис. 4. Адсорбционная эффективность (a) DY132; (b) RR195 на RMA с течением времени контакта, с C_0 - 30, 70 и 100 мг/л

Table 3

Rate constants of pseudo first and pseudo second-order model for adsorption of DY132, RR195 on RMA
***Таблица 3.* Константы скорости модели первого и второго порядка для адсорбции DY132, RR195 на RMA**

Dye	Q_{ep} (mg.g^{-1})	Pseudo-first-order			Pseudo-second-order		
		$k_1 \cdot 10^2$ (min^{-1})	q_e (mg/g)	R^2	$k_2 \cdot 10^2$ (g/(mg·min))	q_e (mg/g)	R^2
RR195	42.12±0.17	5.56	14.25	0.9424	1.48	42.74	0.9984
DY132	54.16±0.22	8.75	15.01	0.9745	1.95	54.95	0.9995

Table 4

Initial rate of dyes adsorption and the time to reach $t_{1/2}$, $t_{0,99}$ on RMA
***Таблица 4.* Начальная скорость адсорбции красителей и время достижения $t_{1/2}$, $t_{0,99}$ на RMA**

Dye	q_e (mg/g)	v_0 (mg·g^{-1}·min^{-1})	$t_{1/2}$ (min)	$t_{0.99}$ (min)
DY132	42.74±0.21	58.9	0.9	92.4
RR195	54.95±0.27	2.0	1.6	156.5

4. Studies of adsorption isotherm on RMA

The influence of the initial concentration of dyes on the adsorption capacity was studied in aqueous solutions at pH 5 using RMA – 1 g/l, the initial concentration (C_0) varied from 30 to 500 mg/l. The parameters H and q were determined in equilibrium and are shown in Fig. 5.

The experimental data were analyzed with both Langmuir and Freundlich isotherm models and are shown in Table 5.

The correlation coefficients R^2 obtained on the Langmuir diagrams were >0.99, which indicates that the Langmuir isotherm model is better suited for adsorption than the Freundlich isotherm model. This is

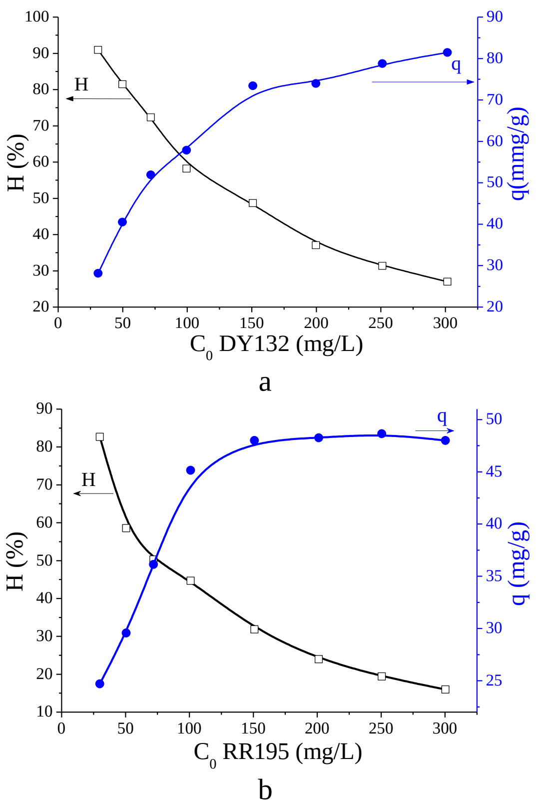


Fig. 5. Effect of initial concentration (a) DY132; (b) RR195 on the adsorption on RMA

Рис. 5. Влияние исходной концентрации (a) DY132; (b) RR195 на адсорбцию на RMA

Table 5

Parameters of DY132 and RR195 adsorption isotherm equations on RMA

***Таблица 5.* Параметры уравнений изотермы адсорбции DY132 и RR195 на RMA**

Dye	Freundlich isotherm			Langmuir isotherm		
	n	K_F	R^2	q_{max} (mg/g)	K_L(L/mg)	R^2
DY132	4.14	1.04	0.9731	84.31	0.08	0.9984
RR195	0.21	$1.52 \cdot 10^{-6}$	0.8935	48.54	0.12	0.9919

single-layer adsorption process, without interaction between adsorbed molecules. The maximum adsorption capacity of DY132 and RR195, respectively, was 84.31 mg/g and 48.54 mg/g.

CONCLUSION

The red mud of the Tan Rai Alumina plant (Lam Dong-Vietnam) after activation by 2M sulfuric acid significantly increased the adsorption capacity as compared to raw red mud. The adsorption process of Direct Yellow132 and Reactive Red195 took advantage at pH = 5, the adsorption kinetics follows the second-order equation, the time to reach the equilibrium of the adsorption process of Reactive Red 195 is longer compared to Direct Yellow132. The adsorption data were well matched to Langmuir isotherm model, the maximum adsorption capacity was found to be 48.54 and 84.31 (mg/g) for Reactive Red 195 and Direct Yellow 132, respectively.

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