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NEW POLYTETRAZOLE SYNTHESIS AND ADSORPTION SURFACE INVESTIGATION BASED ON SULFADIAZINE GRAFTED POLYIMINE

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The present work concentrates on synthesizing and adsorption surface investigation of polytetrazole polymer (P-T) created from polyimine (P-S) grafted with sulfadiazine drug. The prepared polymer is synthesized by mechanochemical polycondensation reaction between dialdehyde aromatic compound and diamine aromatic compound by applying appropriate polar solvent and glacial acetic acid at (78 °C) as dependent on the polymerization reaction. The synthetic route to prepare tetrazole polymer (P-T) consists off multi chemical reaction process, firstly the synthesis of polyimine backbone (P-S) (4-((4'-amino-3,3'-dimethyl-[1,1'-biphenyl]-4-yl)imino)methyl)benzaldehyde) by Schiff base reaction between monomers diamine (*o*-tolidine) and dialdehyde (terephthalaldehyde). Secondly, is the sulfadiazine azid formation, since the conversion of sulfadiazine drug to sulfadiazine azid (4-azido-N-(pyrimidin-2-yl)benzenesulfonamide) occurred by diazonium salt formation of sulfadiazine at very low temperature (0–5 °C) and then reaction with sodium azide (NaN₃) under cool conditions to convert amine group (NH₂) to azide group (N₃), after that sulfadiazine azide was grafted onto the polyimine backbone (P-S) by cyclization reaction process to form polytetrazole (P-T). All prepared products: sulfadiazine azide, polyimine (P-S) and polytetrazole (P-T) were characterized by FT-IR spectroscopy. Finally, the adsorption surface investigations of the prepared polymers (P-S) and (P-T) were studied for adsorption of Brilliant cresyl blue (BCB) dye. All adsorption results were recorded by using an UV-Vis spectrophotometer. Since the polytetrazole (P-T) gave very good adsorption results when applied to (BCB) dye adsorption, there can be suggestion on the polytetrazole polymer applicability in many various environmental and medical applications.

Keywords: polyimine, Schiff base, sulfadiazine, tetrazole, adsorption, Brilliant cresyl blue (BCB) dye

INTRODUCTION

The prepared polymers are many useful in the life sciences to produce medical apparatus, systems of drug delivery, plants, medical applications, biosensors, and application in the adsorption on surface [1]. The production and characterization of polymers explain very good potential in various fields containing medical sciences and biological uses [2]. The molecules of polymers give many features such as drug delivery, catalyst [3]. There are notable polytetrazole with in large kinds of polymers for their flexible characteristics [4]. Newly, the research work about polytetrazoles has extended to contain plans for refining their recital and flexibility by functionalization [5]. Attaching onto the polymer backbone molecules that is bioactive is an method used to produce many functions to the final material [6]. Prepared polymers can exactly regulate their physicochemical properties, controlling the high repeatability [7]. One of the sulfonamides which is sulfadiazine, common

antibiotic sulfonamide drugs, because of its antibacterial characteristics is capable for grafting onto polyimine matrices and compatibility with living tissues [8]. The present work concentrate on the preparation and investigation of surface adsorption of a new polytetrazole derived from sulfadiazine grafted polyimine. This hybrid polymer is synthesized by a combined procedure that includes creating a polyimine backbone and then attaching sulfadiazine moieties using tetrazole links. Adsorption method stands out due to its straightforward operation, inexpensive equipment, and lack of secondary pollutants. However, it faces challenges such as low efficacy, tiny pore diameters, and high cost in typical porous adsorbents [9]. Comprehending the adsorption characteristics of this material is crucial for clarifying its interaction processes with different target molecules and environmental pollutants [10]. The synthetic dye Brilliant cresyl blue (BCB), which is non-azo and has a triphenylmethane structure, gives off a blue hue,

is light –sensitive, and may be found in the food sector, which includes dairy, confections, syrups, and ice cream [11]. Many waste materials were used for dye removal for example (BCB) and methylene blue [12].

EXPERIMENTAL

Materials and method. Commercially accessible chemicals and solvents were required for the procedure. We evaluated the caliber of our produced items using thin-layer chromatography (GF254, Merk, Germany), and we kept track of the UV-induced reactions (254 nm). There were two separate solvent systems used. A and B are, respectively, a 3:1 mixture of *n*-hexane and ethyl acetate. The points of melting were found utilizing the melting point instrument of Stuart SMP3 in open capillary tubes, and they have not been modified. The infrared spectra were produced using a Shimadzu FT-IR spectrophotometer made in Japan.

Synthesis of polymer.

Synthesis of sulfadiazine azide. Dissolve sulfadiazine (0.01 mole) in dilute hydrochloric acid (HCl) solution in a flask, then we slowly added 0.01 mole of the sodium nitrite solution dropwise to the sulfadiazine solution with stirring, maintaining the temperature below 5 °C. This

forms the diazonium salt of sulfadiazine. After that kept the flask containing sulfadiazine solution in an ice bath to cool the reaction mixture at (0–5 °C). After that we added (0.01 mole) of sodium azide (NaN₃) solution to the diazonium salt of sulfadiazine and stirrer the reaction mixture for 5 hours up to white precipitate is formed. Ethanol was used to recrystallize a precipitate. The melting point of product is 265 °C.

Synthesis of Polyimine. A solution of 0.01 mole *o*-tolidine and terephthalaldehyde is dissolved in absolute ethanol in round bottom flask than added 2–3 drops of glacial acetic acid and reflux the reaction at 78 °C, the reaction is completed after four hours and yellow precipitate is formed. After the reaction was finished, the mixture was put into the ice and cold water to cool it down (according to TLC monitoring). Ethanol was used to filter and crystallize a precipitate that was formed from methanol solution.

Synthesis of Polytetrazole. A polyimine (0.5 g) dissolved in DMSO then sulfadiazine azide (1 gm) was added to the reaction mixture and heated for 8 hours at (110 °C), the reaction mixture was stirrer up to reaction is completed, then a yellow precipitate was formed, washed by distilled water and recrystallize by ethanol.

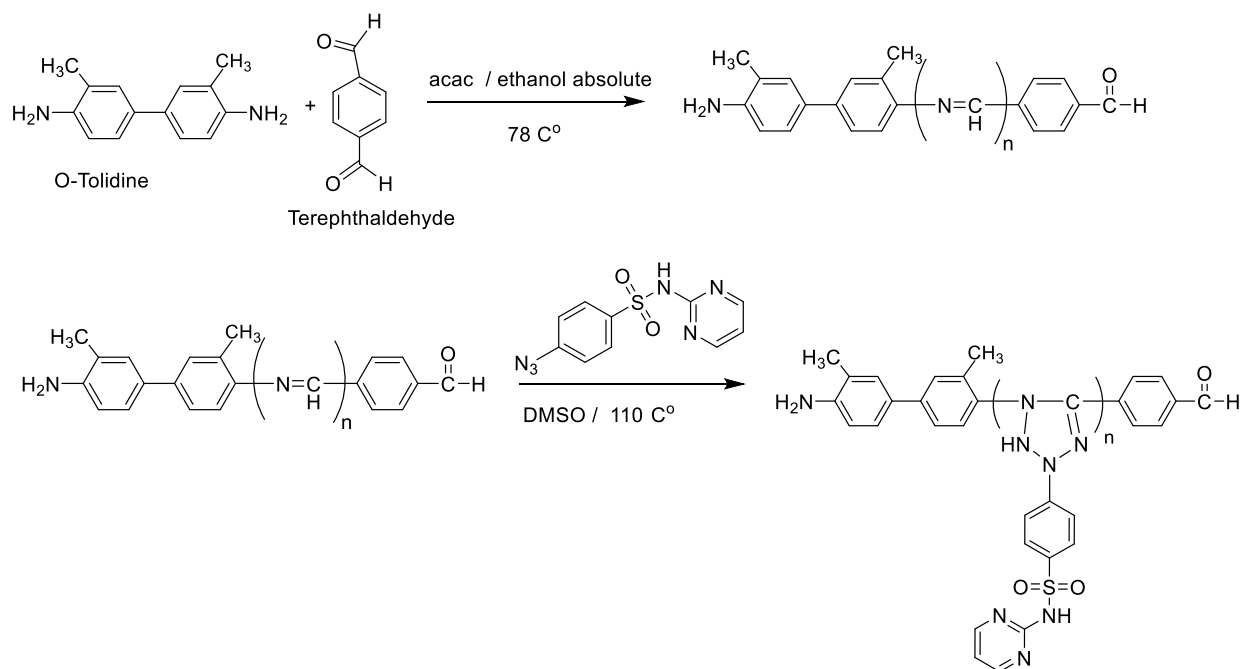


Fig. 1. Synthesis route of polymer

Adsorption experiments. The impact of contact periods (30 min) was examined in all adsorption tests, which were conducted in a series of 25 mL Erlenmeyer flasks holding 10 mL of dye solution with different adsorbent 0.005–0.03 g. Following many contact times and shaking of the solutions, the adsorbents (P-T and P-S) were separated by centrifugation, and the solution was examined using an UV-Vis spectrophotometer.

RESULTS AND DISCUSSION

Chemistry of prepared polymer. The initial step of polymer preparation is the synthesis of sulfadiazine azide which is already prepared by

change the amino group (-NH₂) of sulfadiazine into the azide group (-N₃) by using a diazotization and azidation procedure in the presence of HCl, NaNO₂, and then NaN₃ in the temperature 0–5 °C. In moderate circumstances, the reaction conducted effectively and produced sulfadiazine azide. This transformation was validated by FT-IR spectroscopy, wherein the disappearance of characteristic absorption bands of amino sulfadiazine in which appear in the range 3460–3370 cm⁻¹ and appearance of new peaks at 2132–2106 cm⁻¹ corresponding to sulfadiazine azide confirmed the successful conversion.

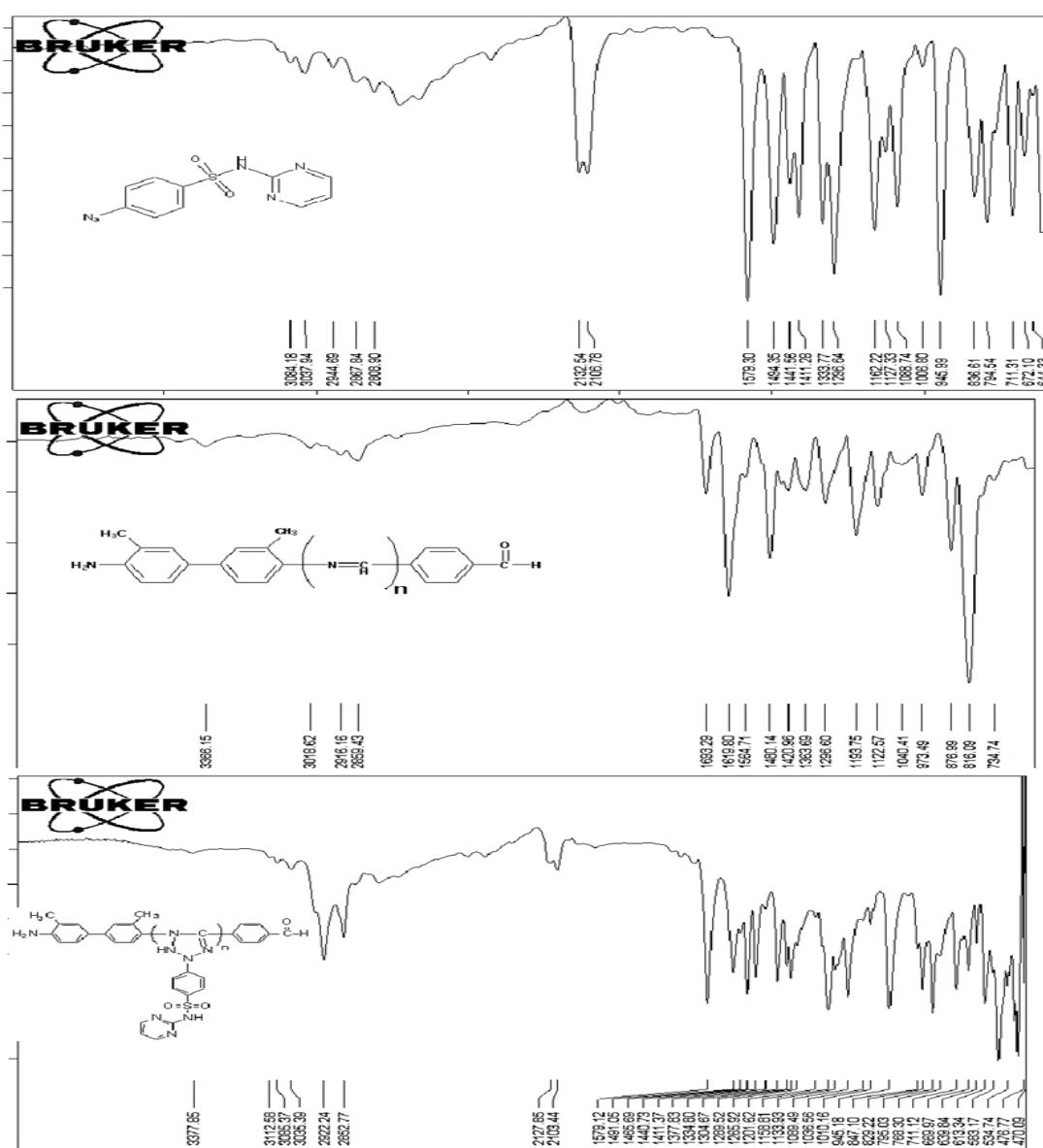


Fig. 2. Mixed FT-IR spectrum of prepared polymer

The polyimine is prepared by the condensation reaction between the terephthalaldehyde and *o*-tolidine by Schiff base formation. The capability of this synthetic technique to create successfully the polyimine product via the Schiff base reaction between *o*-tolidine and terephthalaldehyde shows how versatile is it for the production of polymeric materials. A high molecular weight polymer is created when the polymer chain is extended due to the Schiff base connection that forms between the amino group of *o*-tolidine and the aldehyde group of terephthalaldehyde. This transformation was investigated by FT-IR spectroscopy, wherein the disappearance of characteristic absorption bands of amino *o*-tolidine in which usually appears in the range 3480–3396 cm^{-1} and disappearance of carbonyl terephthalaldehyde in which usually appears in the range 1682–1675 cm^{-1} and appearance of new strong bands were observed around 1693–1619 cm^{-1} , corresponding to the stretching vibrations of the imine (-C=N-) functional group. Additionally, absorption peaks around 2916–2859 cm^{-1} confirmed the presence of aliphatic C-H stretching vibrations in the polymer backbone while aromatic C-H stretching vibrations appearance at 3018 cm^{-1} .

Finally, the the polyimine backbone was functionalized with sulfadiazine moieties via cyclization reaction to form tetrazole rings between the polyimine and sulfadiazine azid. This transformation was validated by FT-IR spectroscopy, where in the reducing appearance of characteristic absorption bands of azide and imine groups and the appearance of new peaks at 3112–3065 cm^{-1} corresponding to C-H band in the tetrazole ring confirmed the successful conversion. All characterized peaks are showed in the (Fig. 2) mixed FT-IR spectrum of prepared polymer.

Adsorption result. The (BCB) dye was subjected to equilibrium sorption on polyimine which is polymer-Schiff (P-S) and polytetrazole which is polymer-tetrazole (P-T) by contacting 0.01 g of the adsorbent with 10 ml of varying concentrations ranging from 10 to 60 mg/L for a duration of 25 minutes. After the mixture was filtered, an UV-Vis Spectrophotometer was used to determine the filtrate's residual concentration. Using the formulas published by Vanderborght and Van Griekenm, the quantity of adsorbed (mg/g) was determined [13]:

$$Q_e = \frac{(C_0 - C_t)V}{W}$$

where Q_e = how much solute has been adsorbed out of the mixture. The variables V stands for volume of the adsorbate, C_0 for concentration prior to adsorption, C_t for concentration following adsorption, and W for the adsorbent's weight in grams. Using the formulas in Equation, the % sorption was calculated to estimate the removal efficiency [5]:

$$\% \text{Removal} = \frac{(C_0 - C_t)}{C_0} \times 100.$$

Impact of Interaction Duration. A study was conducted at 25 °C to examine the impact of contact time on the adsorption of BCB dye on P-T and P-S. The amount of adsorption removal varies over time, as seen in Fig. 1. The findings demonstrate that the dye solution adsorption on P-T surface reaches an equilibrium stage after 15 min and thereafter decreases, whereas the dye solution adsorption on P-S surface reaches its maximum at 25 min as a result of the attractive forces that have generated between the dye and surface. Desorption happens after the equilibrium stage [14].

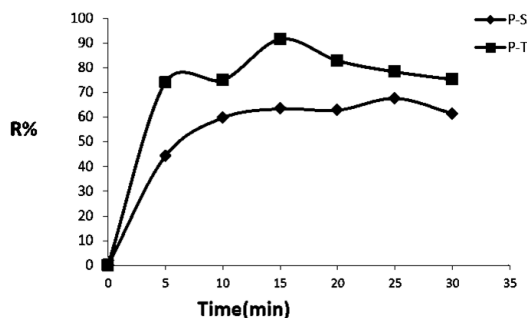


Fig. 3. Impact of contact duration on (BCB) dye adsorption on P-T and P-S surfaces

Effect of amounts of adsorbent. The effect of the weight of the adsorbent P-T and P-S surfaces on the adsorption of the BCB dye was studied by changing the weight of the adsorbent surface within the range of 0.005–0.03 g while keeping the dye concentration at 60 mg/L, temperature at 25 °C, and shaking time constant. Fig. 2 shows

the relationship between the removal percentage and the P-T and P-S weight.

The percentage of adsorption increases with increasing weight up to 0.01 g for the P-T and 0.015 g for the P-S and then decreases. The reason is due to the increase in the active sites or the increase in exchange sites on the surfaces [15].

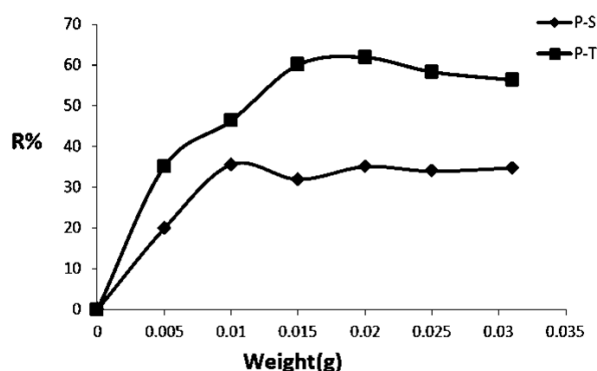


Fig. 4. Impact of adsorbent quantity on BCB dye adsorption by P-T and P-S

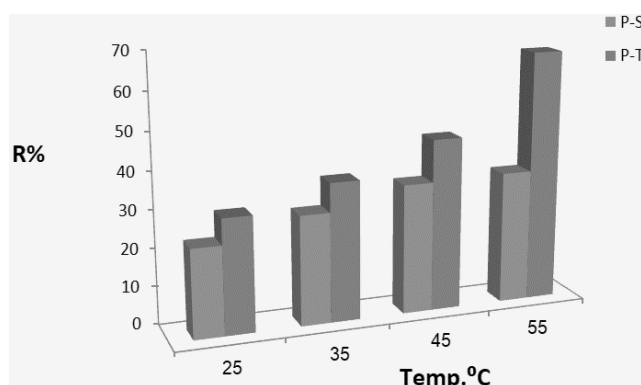


Fig. 5. The impact of changing temperature on the percentage of adsorption of dye BCB on the surfaces of P-T and P-S

Effect of temperature. The impact of temperature on the adsorption of BCB dye was studied in a range of 25–55 °C on the surface of the P-T and P-S. Fig. 3 shows the results. The indicating of results that the amount of adsorption increases with increasing temperature, because with increasing temperature, the effective sites for adsorption increase on the adsorbent surfaces and the porosity and size of the pores on the surface increase. Also, increasing the temperature reduces the viscosity of the dye solution, increases the kinetic energy of the dye molecules, and increases their diffusion on the surface [16, 17].

Thermodynamic parameters. Using the equilibrium constant K_{ads} , the thermodynamic parameters for the adsorption of BCB dye on P-T and P-S, such as ΔH , ΔS , and ΔG , were

determined. The energy change in of Gibbs should be calculated from the equation:

$$\Delta G = -RT \ln K_{ads}$$

since the $K_{ads} = \frac{Q_e}{C_e}$.

The enthalpy and entropy of adsorption may be obtained from the equation:

$$\ln K_{ads} = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$$

Making a plot between $\ln K_{ads}$ versus $(1/T)$ will form a straight line with a slope $-\Delta H/R$ and intercept $\Delta S/R$ as shown in Fig. 4.

The quantitative thermodynamic data of BCB dye on the adsorbent surfaces P-T and P-S are provided in Table 1. Since the BCB dye's ΔH value is positive, the adsorption process is

endothermic [18, 19]. The adsorption process consider spontaneous [20] than the negative value of ΔG . Conversely, ΔS for dye has a positive

value, indicating that the randomness of the entire system is produced by molecular interactions.

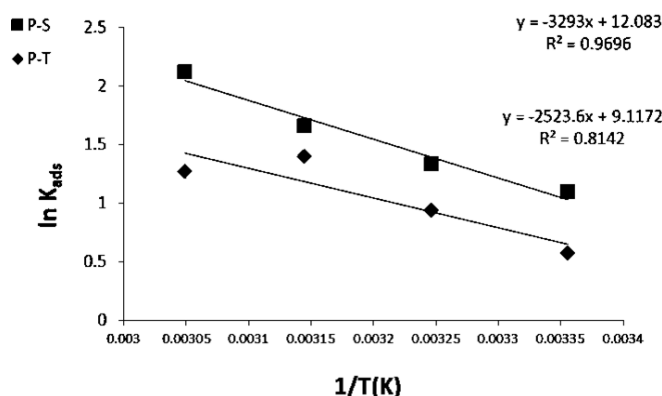


Fig. 6. Arrhenius equation for the adsorption of dye BCB dye on the surfaces of Polymer-Schiff (P-S) and Polymer-Tetrazole (P-T)

Table 1. The BCB dye’s thermodynamic functions on the adsorbent surfaces are ΔG , ΔS , and ΔH

T/K	1/T	K_{ads}		$-\Delta G / (\text{kJ mole}^{-1})$		$\Delta H / (\text{kJ mol}^{-1})$		$\Delta S / (\text{J mol}^{-1}\text{K}^{-1})$	
		P-S	P-T	P-S	P-T	P-S	P-T	P-S	P-T
298	0.003356	1.772482	2.987857	1.41812	2.71184				
308	0.003247	2.552871	3.780345	2.39995	3.40527	20.98	27.37	75.80	100.45
318	0.003145	4.04349	5.238766	3.69375	4.37845				
328	0.003049	3.560477	8.283249	3.46299	5.7655				

Adsorption isotherm. Two adsorption isotherm models, including the Freundlich and Langmuir isotherms, were used to research the maximum sorption capacity of the adsorbent and to investigate the link between P-T, P-S surface, and BCB dye at equilibrium. The results of adsorption were analyzed according to the linear Langmuir isotherm [21], as shown in Fig. 5.

$$\frac{1}{Q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 \cdot b \cdot C_e}$$

where C_e = the concentration of equilibrium of adsorbate (mg/L^{-1}), Q_e = the metal amount of adsorbed per gram of the adsorbent at equilibrium (mg/g). Q_0 = maximum monolayer coverage capacity (mg/g) b = Langmuir isotherm constant (L/mg).

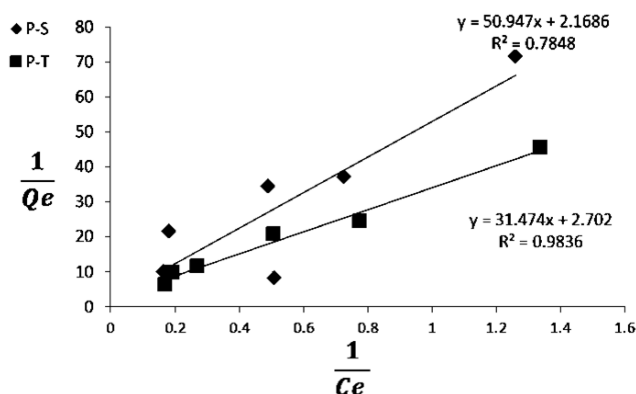


Fig. 7. Langmuir isotherm of dye BCB dye on the surface of the polymer-Schiff (P-S) and polymer-tetrazole (P-T)

The values of the correlation coefficient (R^2) were 0.9836 and 0.7848 for the P-T and P-S, respectively, which indicates that the Langmuir equation is suitable for the results of the adsorption of the BCB dye on the two surfaces.

The Freundlich model of linear form is:

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e$$

where C_e is the dye equilibrium concentration in solution (mg L^{-1}) and Q_e is the equilibrium dye concentration sorbed in sorbent ($\text{mg}\cdot\text{g}^{-1}$). The empirical value $1/n$, which relates to sorption intensity and fluctuates with material heterogeneity, and the Freundlich constant K_f , which is connected to sorption capacity [22].

The values of the (R^2) is about 0.4490 when using the P-S surface, which indicates that this equation is not suitable for the results of dye adsorption on the P-S surface, while the value of (R^2) was 0.9673 when used P-T, which indicates the suitability of the Freundlich equation for the results of adsorption on the surface of the P-T, as in Fig. 6.

The Freundlich constants were calculated using the P-T as an adsorbent surface and it was found that $n = 1.16$, which indicates that the physical adsorption of BCB dye on P-T surface. The value of the Freundlich constant was $K_f = 0.029$.

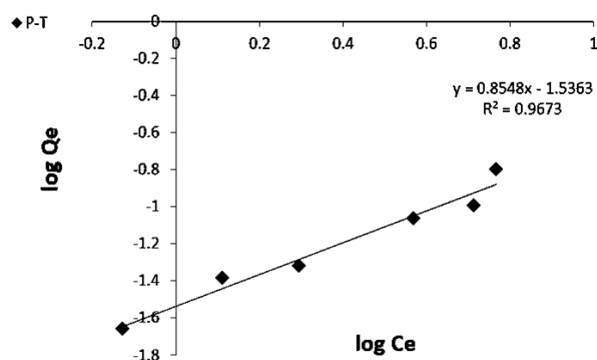


Fig. 8. Freundlich isotherm of dye BCB dye on the surface of the polymer- tetrazole (P-T)

CONCLUSION

The synthesis and investigation of the adsorption surface of the novel polytetrazole derived from sulfadiazine grafted polyimine is a notable development in polymer chemistry and materials science. Using a systematic approach, we have successfully produced and examined a hybrid polymeric material with tailored properties that may be used for environmental remediation, catalysis, and delivery of drugs. The first producing is the synthesis of polyimine backbone by Schiff base reaction and then linking bioactive molecule sulfadiazine by cyclization reaction to produce polytetrazole polymer. The investigation of adsorption surface of prepared polymer gave many benefits, since the important information about the created polymer interactions with aim substances and environmental contaminants was supplied by the investigation of adsorption surface.

The preparation and investigation of surface adsorption on synthesized polymer gives large advancement in the field of polymeric materials and lets us to challenge critical matters in the subjects like environmental sustainability, healthcare, and materials science, thus helping the progress of scientific understanding and technological progression.

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Новий синтез політетразолу та дослідження адсорбційної поверхні на основі полііміну, з прищепленим сульфадіазином

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Ця робота зосереджена на синтезі та дослідженні адсорбційної поверхні політетразольного полімера (P-T), створеного з полііміну (P-S), щепленого сульфадіазиним препаратом. Отриманий полімер синтезують шляхом механохімічної реакції поліконденсації між ароматичною сполукою діальдегіду та ароматичною сполукою діаміну шляхом застосування відповідного полярного розчинника та крижаної оцтової кислоти при (78 °C) залежно від реакції полімеризації. Синтетичний шлях отримання полімера тетразолу (P-T) складається з процесу багатьох хімічних реакцій, по-перше, синтезу поліімінової основи (P-S) (4-(((4'-аміно-3,3'-диметил-[1,1'-]біфеніл)-4-іл)іміно)метил)бензальдегід) реакцією основи Шиффа між мономерами діамін (o-толідін) і діальдегід (терефталальдегід). По-друге, це утворення сульфадіазинового азиду, оскільки перетворення препарату сульфадіазинового ряду на сульфадіазинний азид (4-азидо-N-(піримідин-2-іл)бензолсульфонамід) відбувається шляхом утворення діазонієвої солі сульфадіазину при дуже низькій температурі (0–5 °C), а потім реакція з азидом натрію (NaN₃) у прохолодних умовах для перетворення аміногрупи (NH₂) на азидну групу (N₃), після чого азид сульфадіазину був прищеплений до поліімінової основи (P-S) за допомогою процесу реакції циклізації з утворенням політетразолу (P-T). Усі отримані продукти: сульфадіазин азид, поліімін (P-S) і політетразол (P-T) були охарактеризовані методом FT-IR спектроскопії. Дослідження поверхні готових полімерів (P-S) і (P-T) були виконані для адсорбції барвника Brilliant Cresyl Blue (BCB). Усі результати адсорбції реєстрували за допомогою спектрофотометра UV-Vis. Оскільки політетразол (P-T) дав дуже хороші результати при застосуванні для адсорбції барвника (BCB), можна висловити припущення щодо придатності полімера політетразолу в багатьох різноманітних екологічних і медичних застосуваннях.

Ключові слова: поліімін, основа Шиффа, сульфадіазин, тетразол, адсорбція, барвник Brilliant Cresyl Blue (BCB)

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