



## The Atrazine Removal with the Polyaniline Coated Rice Husk as a Cheap Adsorbent

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Adsorption,  
Atrazine,  
Polyaniline,  
Rice husk

### Abstract

In this study, a composite of rice husk (RH) modified with polyaniline (PANI) was produced and its potential as an adsorbent in removal of atrazine was investigated. Within the scope of the study, the effects of contact time (0-480 min), initial pH (3.5-9.5), initial atrazine concentration (2-25 mg/L), and PANI/RH amount (0-1.6 g) on treatment efficiency were examined. The optimum treatment efficiency for atrazine (25 mg/L) was found as 58.3% under 120 min., 5.4 of pH, and 1.0 g/50 ml of adsorbent dosage, and at this condition, adsorption capacities ( $q_t$ ) was calculated as 0.58 mg/g. Furthermore, when the initial atrazine concentration was raised from 2 to 25 mg/L, the removal efficiencies decreased from 81.1 to 60.4 %, but the adsorption capacities ( $q_t$ ) increased from 0.067 to 0.629 mg/g. In addition, the efficiency of the adsorption process was evaluated by applying Langmuir and Freundlich isotherm models. Among the performed isotherm models, Freundlich isotherm provided the best correlation for atrazine and the Freundlich constant related to the sorption capacity was calculated as 2.02 mg/g at an initial pH of 5.4 for the 2.0-25 mg/L atrazine at 25 °C. Raw-PANI/RH and used-PANI/RH composites were characterized with FTIR, XRD, and SEM analysis.

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## 1 INTRODUCTION

Atrazine has been frequently detected in surface and/or ground waters because it is widely used (70,000–90,000 tons/year worldwide) against broadleaf and grassy weeds as a triazine herbicide with the molecular formula  $C_8H_{14}ClN_5$ . Atrazine is carcinogenic, has endocrine-disrupting effects, and has high mobility in aqueous media, even low water solubility (33 mg/L at 20 °C). Due to the stable structure, the long residence time in the environment (30–100 days of half-life), and toxicity of atrazine, it should be removed from water sources [1]. Until now, Physico-chemical (adsorption, chemical oxidation, etc.) and biological treatment systems widely employed for atrazine removal [2]. Atrazine is resistant to oxidation by ozone [3]. Furthermore, the studies that depend on ozone coupled with  $H_2O_2$ , Mn(II) or ultraviolet (UV) radiation [4, 5] showed the difficulties of atrazine degradation.

This compound has been removed by photodegradation [6], UV/ $H_2O_2$ [7], Fenton [8], electrooxidation (EO) with Pt, metal oxides and BDD (Boron Doped Diamond) electrodes [9], and Electro Fenton (EF) with BDD [10–12]. In these studies, it is observed that a by-product, cyanuric acid (2,4,6-trihydroxy-1,3,5-triazine) is formed. It is very stable and inhibits full mineralization. These studies showed that only atrazine could be slowly degraded by BDD anode at high voltage. However, especially high radical concentration can increase the toxicity of wastewater [13, 14], and operational costs can be high due to the chemical consumed. Biological treatment technology, including microbial remediation, phytoremediation, and plant-microbial remediation, is a frequently used technique for atrazine degradation. However, the high toxicity of atrazine and the long-term requirement of biological treatment limit the applications of these systems [9].

Another technique used for the treatment of atrazine in literature is adsorption, although it is an effective method for only batch and low concentration. Especially activated carbon and biochar are the most used adsorbents in the treatment of atrazine by adsorption[15–17]. However, the difficulties in the adsorption process, such as the regeneration problems and the high cost, restrict its application. Thus, scientists have focused on finding a promising alternative that should be inexpensive and provide higher removal efficiencies. Recently, it has been found that the usage of polymeric materials and inorganic materials for the production of composite material presents a combination of both materials' advantages [18] in the production of new adsorbents.

In this study, a composite of rice husk (cheap natural material) and polyaniline (polymeric substance providing high treatment efficiency) was produced, and its potential as an adsorbent in the removal of atrazine was investigated. Within the scope of the study, the effects of the contact time, initial pH, initial atrazine concentration, and PANI/RH amount on treatment efficiency were examined, and also Langmuir and Freundlich isotherms obtained from equilibrium data were examined to evaluate the efficiency of the adsorption process.

## 2 MATERIAL AND METHODS

### 2.1 Experimental procedure and instrumentation

Atrazine was analyzed by high-pressure liquid chromatography (HPLC, Agilent) equipped with an Agilent C18 column (150 mm × 4.6 mm, 5  $\mu$ m). The mobile phase was selected as a mixture of acetonitrile and water (50:50, v/v) with a flow speed of 0.8 mL/min. A UV detector was used at 220 nm. A calibration graph was prepared between 0-25 mg/L of concentration. All the used chemicals supplied by Merck were of an analytical grade and used without further purification. PANI/RH composite's chemical and structural properties was analyzed using Fourier transform infrared (FTIR, Perkin Elmer Frontier FT-IR LR64912C) spectra with KBr pellets in the infrared region 4000-400  $cm^{-1}$  and X-ray diffraction analysis (XRD, PANalytical X'Pert Pro Materials Research Diffractometer) with Cu-K $\alpha$  radiation at a 2 $\theta$  angle values between 10° and 90° in 1o  $min^{-1}$ . PANI/RH composites' surface morphologies were characterized using scanning electronic microscopy (SEM, Zeiss Supra VP 40).

### 2.2 Preparation of Polyaniline/RH composite

For the preparation of composite materials, 20 g of the rush husk (used as received without any pretreatment) was added into 2000 ml HCl (1 M) solution, and the solution was mixed at 500 rpm [19]. Then, 43.640 g ammonium persulfate (APS, 0.1 M) was added into the HCl solution at room temperature, and 18.62 ml of aniline monomer (0.1 M) was injected slowly. The polymerization was carried out at room temperature for 2 h, and to remove the unreacted monomers and oxidants, the dark blue final product was washed thoroughly with 5000 ml of deionized water. Finally, the product was dried at 60 °C for 24 h. Thus, the molar ratio of APS and aniline was adjusted to 1.0.

Batch adsorption runs were carried out to find optimum adsorption conditions in a shaker. 50 ml of synthetic solutions were prepared, and the pH was adjusted using 1N H<sub>2</sub>SO<sub>4</sub> or 1N NaOH by using a pH meter. The temperature was kept constant at 25 °C during the experiments. The removal efficiencies ( $E_R$ , %) have been calculated according to Equation 1:

$$E_R = \left[ \frac{C_0 - C_f}{C_i} \right] \times 100 \quad (1)$$

Adsorption capacities ( $q_t$ ; mg/g) was determined by the following formula (Eqn. 2):

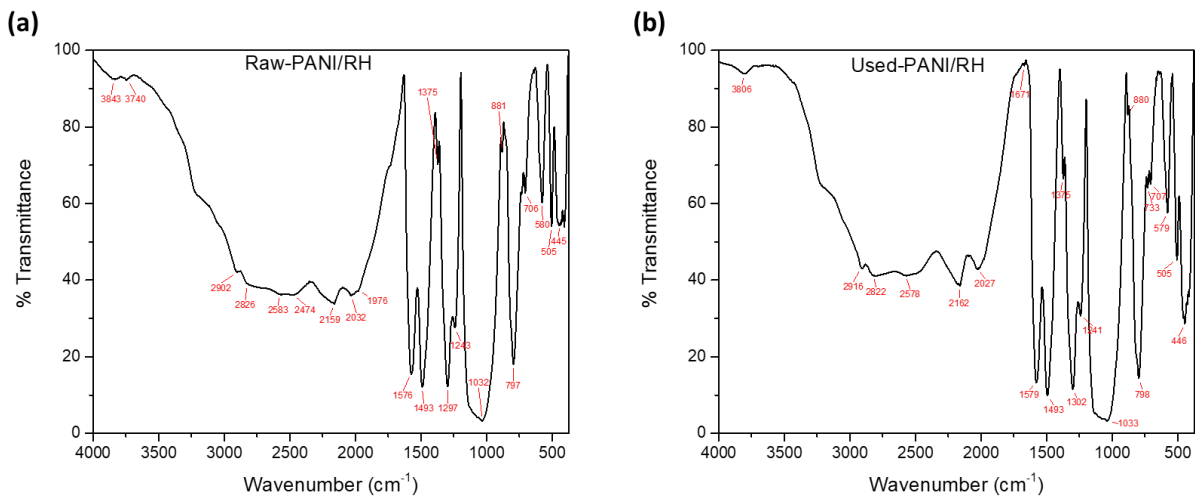
$$q_t = \frac{(C_0 - C_t)V}{m} \quad (2)$$

where,  $C_0$  and  $C_t$  (mg/L) denote the initial and equilibrium concentrations of atrazine in solution,  $V$  (L) shows the sample volume, and  $m$  (g) is the amount of the adsorbent.

### 3 RESULT AND DISCUSSION

#### 3.1 Characterization of Raw and Used PANI/RH

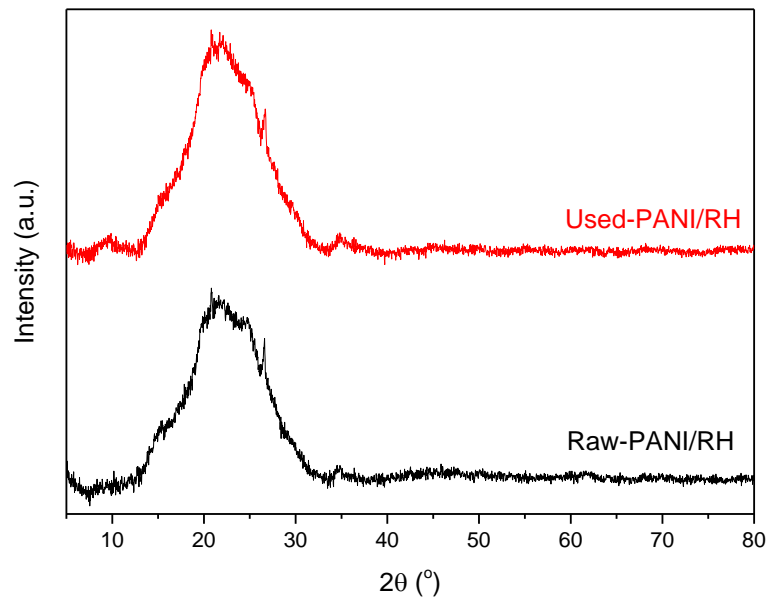
The FTIR spectrum of synthesized PANI/RH composite (Raw-PANI/RH) and PANI/RH composite used in the removal of atrazine (Used-PANI/RH) is shown in Figure 1. According to literature, the FTIR bands for PANI specific to N–H bond vibrations (3370, 3200, 3023 cm<sup>-1</sup>), C–H vibrations (2961, 2921, 2862, 1373, 995, 971, 909 cm<sup>-1</sup>), C=N bond vibrations (1650 cm<sup>-1</sup>), C–N bond vibrations (1310, 1255 cm<sup>-1</sup>), and C=C bond vibrations (1597, 1515, 1496, 1450 cm<sup>-1</sup>) are observed[20]. In addition, RH RH includes the main functional groups is alcohol, phenol, and carboxylic acid (3600-3000 cm<sup>-1</sup>), aliphatic C-H (2915-2845 cm<sup>-1</sup>), ketone (2299 cm<sup>-1</sup>), carbonyl (1716 cm<sup>-1</sup>), aromatic hydrocarbon (1601 cm<sup>-1</sup>), and Aromatic C-H (791 cm<sup>-1</sup>)[15]. As this literature information supports, there are characteristic peaks of PANI and RH in the FTIR spectrum of the Raw-PANI/RH composite given in Figure 1a, and the composite was produced successfully by polymerization of PANI. In addition, newly emerged peaks at 1671 and 733 cm<sup>-1</sup> in the FTIR spectrum of the used-PANI/RH composite in Figure 1b are the peaks responsible for the atrazine-specific Triazine group and the C-H stretching on the aromatic ring[21], and these results support the adsorption of atrazine to the composite.



**Figure 1.** FTIR spectra of (a) Raw-PANI/RH composite and (b) Used-PANI/RH composite

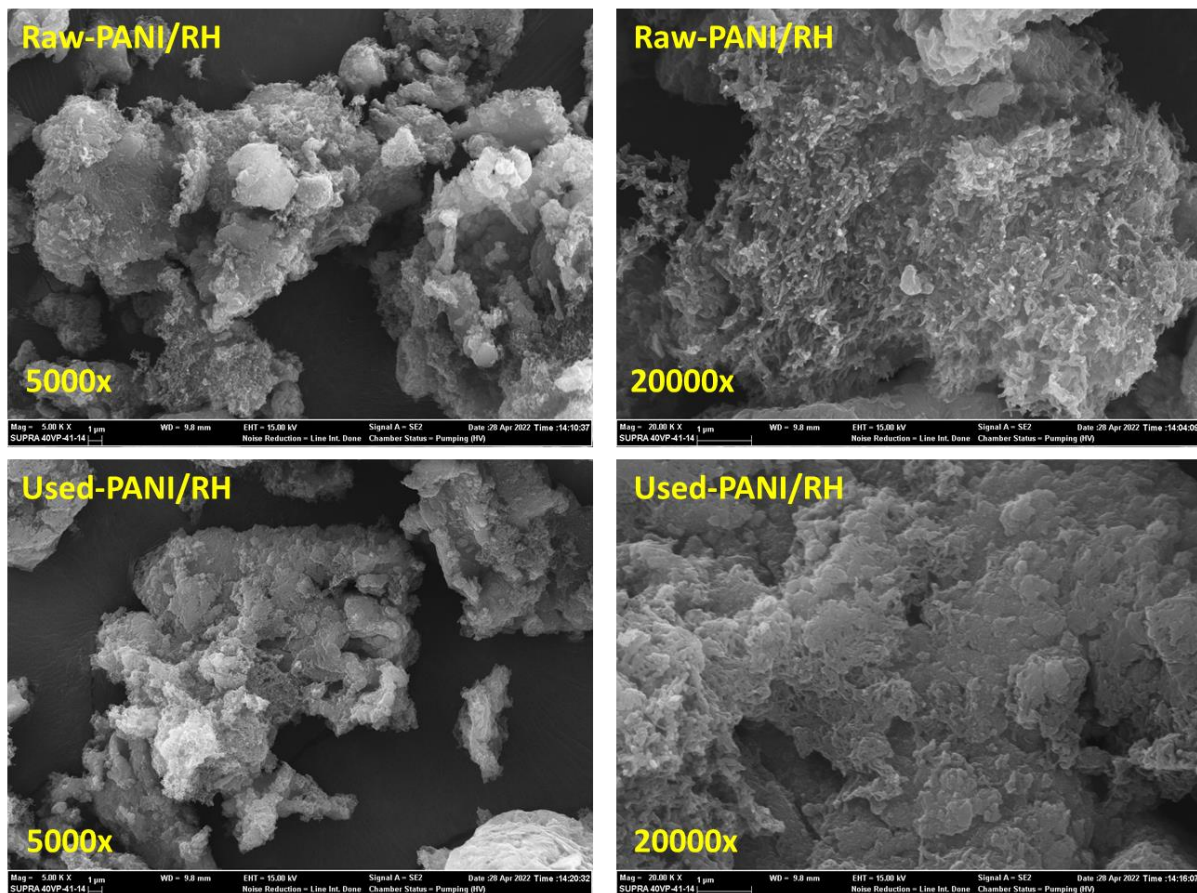
XRD patterns of raw-PANI/RH and used-PANI/RH composites are shown in Figure 2. the XRD pattern of both raw-PANI/RH and used-PANI/RH composites does not show a sharp peak, and this smooth XRD pattern can be indicated the amorphous nature of polyaniline[22]. Thus there is no crystallinity occurred during the polymerization of aniline and it indicates the uniform growth of polyaniline onto the rice husk. According to the literature, the XRD pattern of rice husk shows peak diffraction at  $2\theta = 16^\circ$ ,  $22^\circ$ , and  $34^\circ$ , and also for PANI, the characteristic peaks appear at  $15.3^\circ$ ,  $20.4^\circ$ , and  $26.28^\circ$ [22, 23]. As can be seen from Figure 2, there is a wide peak between  $15-34^\circ$  for raw and used PANI/RH composite, and this range is the region where characteristic XRD patterns occur for both PANI and RH. Therefore, the peaks for both composite components are overlapped. However, the peaks at  $26^\circ$  and  $34^\circ$  are PANI and the rice husk specific, respectively. Also, the peak in used-PANI/RH composite is sharpened after atrazine adsorption compared to raw-PANI/RH composite. Also, it is

explained that the wide-angle XRD pattern implies an amorphous-like pattern that corresponded to the existence of large porous in the material[22]. SEM images in Figure 3 support XRD results and raw-PANI/RH surface porosity decreases than that of PANI/RH composite surface after atrazine adsorption.



**Figure 2.** XRD patterns of (a) Raw-PANI/RH composite and (b) Used-PANI/RH composite

Figure 3 is shown SEM images of the raw and used PANI/RH composites. Raw-PANI/RH composite powder surface includes porous structures like fiber and reticular. After atrazine adsorption, used-PANI/RH composite surface has become smoother than raw-PANI/RH surface as a result of the filling by the atrazine of PANI/RH pores.



**Figure 3.** SEM images of raw-PANI/RH and used PANI/RH composites

### 3.2 The Effect of Contact Time

The effect of the contact time (0-480 min) was evaluated, and the obtained results are shown in Fig. 4. The removal efficiency increased rapidly due to the abundant availability of active binding sites on the sorbent in the initial stages (up to 60 min.), and the sorption became less efficient with gradual occupancy of these sites between 60-90 min and finally, the maximum removal was realized at 120th min. The contact time between 120-480 min shows no significant differences in the removal efficiency and adsorption capacity. In addition, although the effect of the contact time has been studied for a very long time (480 min), as seen in Figure 1, no desorption has occurred. The results showed that the removal mechanism is very fast. These results correspond with study results in the literature [24]. In the experiments performed in the next parts of the study, 120 minutes was taken as the optimum contact time.

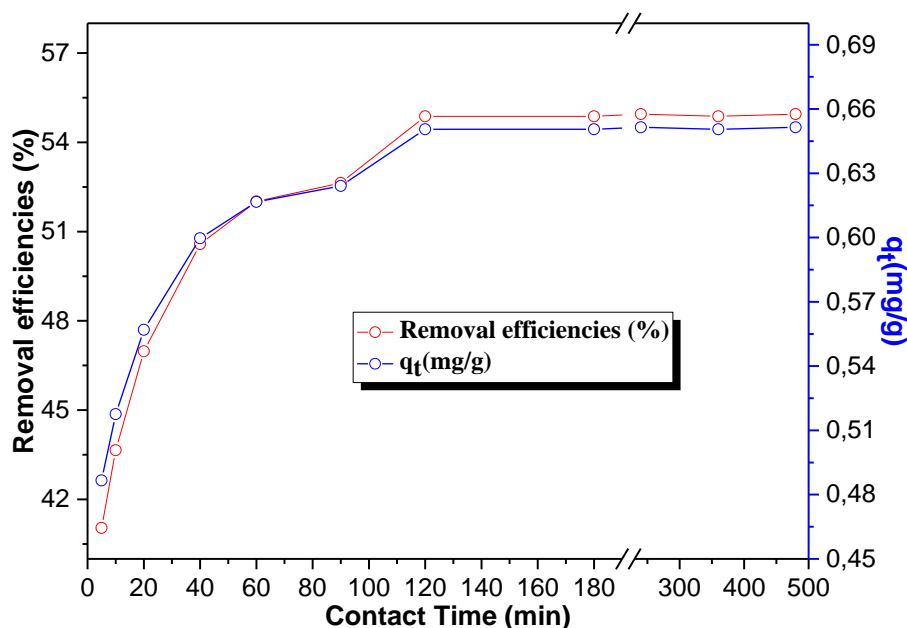


Figure 4. The effect of contact time ( $C_i=25$  mg/L, pH=5.4,  $m=1.0$  g,  $V=50$  ml, 250 rpm)

### 3.3 The Effect of Initial pH

The initial pH of atrazine solution was measured as 5.4, and the effect of initial pH was investigated by the arrangement of solution pH between 3.5-9.5 (Fig.5). As seen in Figure 5, the removal efficiencies of 37.9 (pH 5.4) and 40.0 % (pH 8.5) were obtained during these experiments, and only 2.1% of the difference between the maximum and minimum removal efficiencies was measured. It is reported that atrazine has two slightly different pKa values, 1.60 and 1.95, corresponding to protonation on heteroatoms. It means that, at pH close to these pKa values, half of atrazine is in cationic form, while the other half is in the non-ionic form. So, at other pH values studied in this study, all atrazine molecules occurred in their neutral form. Therefore, the independence of the adsorption efficiency on pH suggested how the interaction between atrazine molecules and polymer surface functional groups was mainly due to van der Waals forces and H-bonds, as already reported in the literature for atrazine and other adsorbent materials [25]. Although the highest treatment efficiency was obtained at pH 8 as according to the literature [16], considering the costs required to adjust the pH, it was clear that the optimum treatment efficiency was obtained at the natural pH value (5.4). Therefore, in the next parts of the study, the pH of 5.4 was taken as the optimum initial pH.

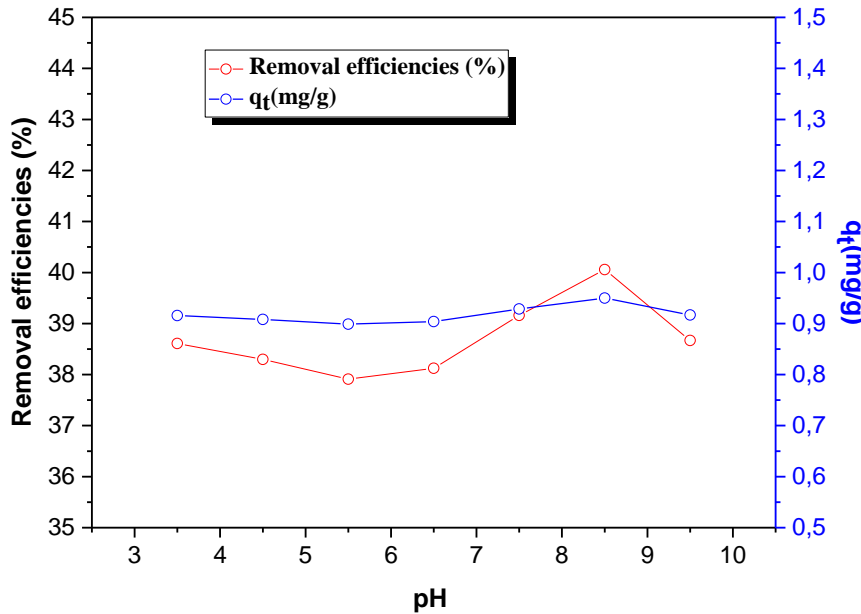


Figure 5. The effect of initial pH ( $m=0.5$  g,  $C_i=25$  mg/L,  $V=50$  ml,  $t=120$  min, 250 rpm)

3.4 The Effect of Initial Atrazine Concentration

The effects of different initial atrazine concentrations ( $C_i$ ) were studied (Fig. 6). When  $C_i$  is increased from 2 to 25 mg/L, the removal efficiencies decreased from 81.1 to 60.4 %, but the  $q_t$  increased from 0.067 to 0.629 mg/g. As expected, increasing  $C_i$  enhances the interaction between atrazine and PANI/RH, which causes an increase of the  $q_t$ . However, the surface area of the adsorbent has a capacity, and the atrazine removal efficiency decreased with an increase in  $C_i$ . This was likely due to insufficient surface area where the adsorption takes place.

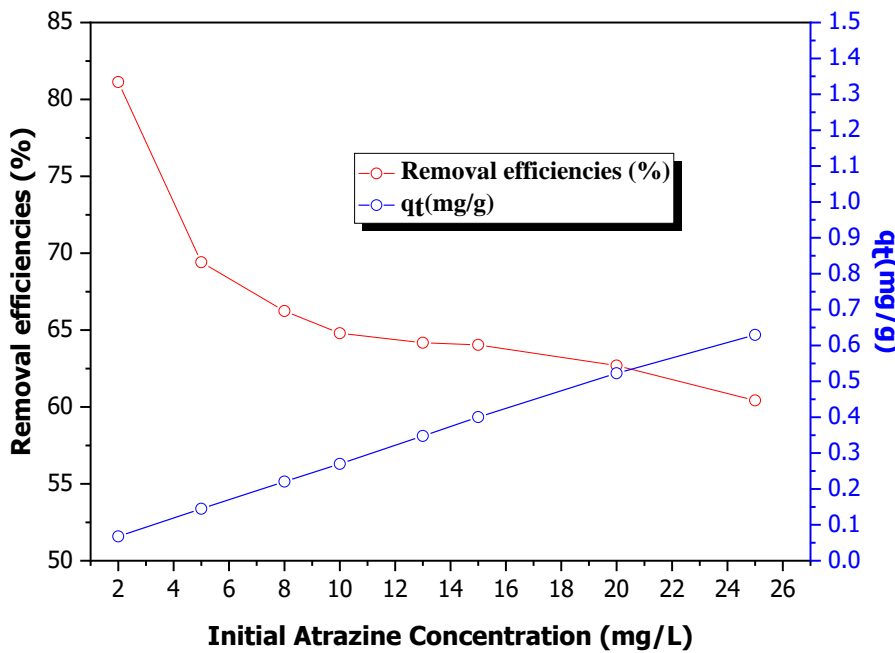


Figure 6. The effect of initial Atrazine concentration (pH=5.4,  $m=1.2$  g,  $V=50$  ml,  $t=120$  min, 250 rpm)

3.5 The Effect of Adsorbent Dosage

The effect of adsorbent dosage was tested with an addition of 0.02, 0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.8, 1.0, 1.2, 1.4, 1.5, and 1.6 mg to 50 ml of 25 mg/L atrazine solution as seen in Figure 7. At the end of the experiments, removal efficiencies were obtained as 5.8 %, 9.7 %, 11.6 %, 19.4 %, 26.7 %, 33.2 %, 38.3 %, 41.6 %, 48.8 %, 54.9 %, 58.3 %, 61.7 %, 63.8 %, and 64.0 %, respectively. The optimum adsorbent dosage of 1.2 g/50 mL is



selected because it is the point located on the linear regression curve (Fig. 7) and ensures maximum removal efficiency.

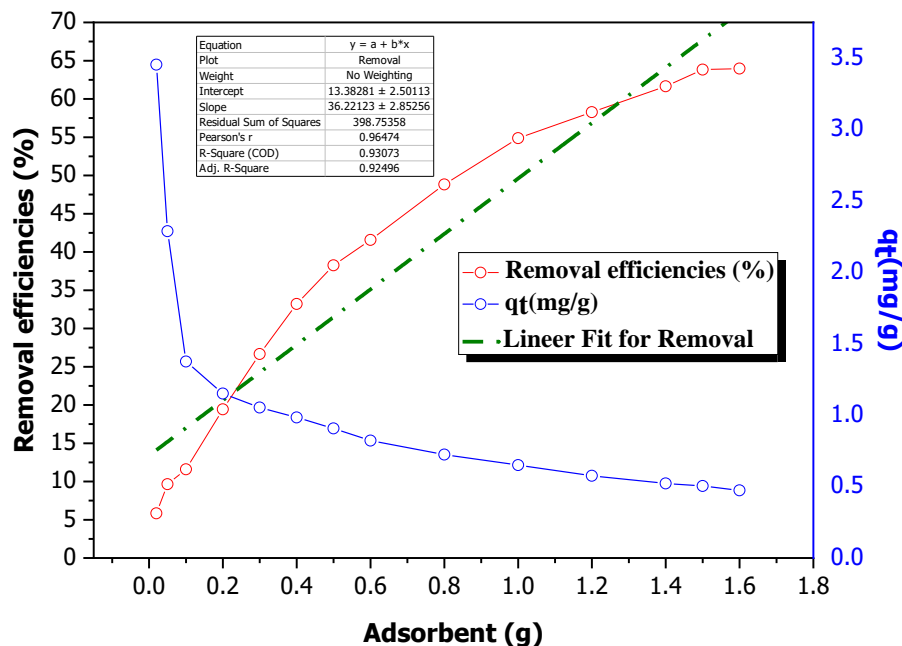


Figure 7. The effect of PANI/RH amount (pH=5.4,  $C_i=25$  mg/L,  $V=50$  ml,  $t=120$  min, 250 rpm)

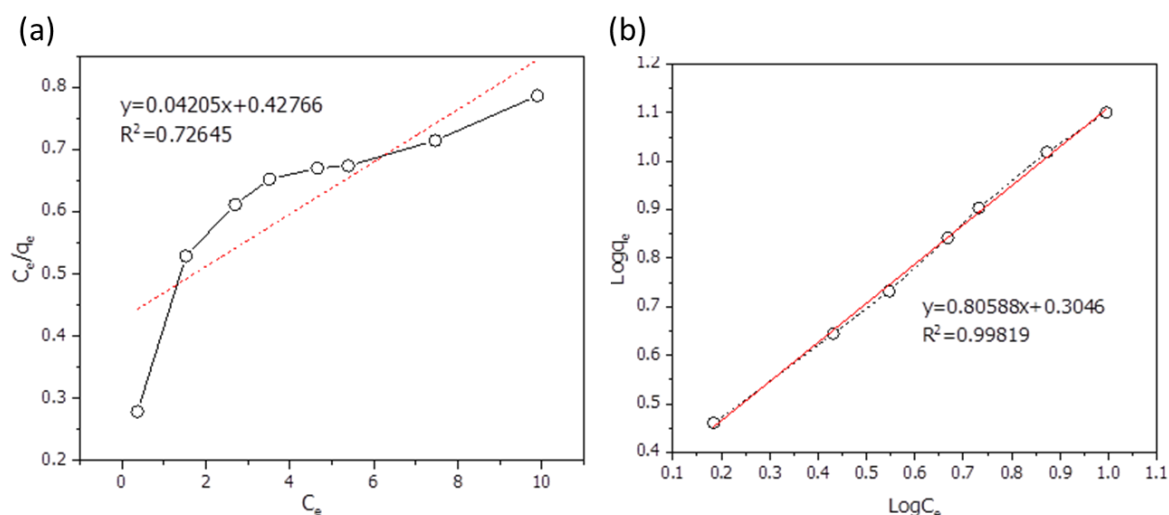
### 3.6 Isotherm Studies

The analysis of the adsorption isotherm data is an important step in finding a suitable model to be used for adsorption system design. In this study, four isotherm models which are the Langmuir and Freundlich, were used. Isotherm calculations have been performed in range of the 2.0-25.0 mg/L atrazine concentrations. The Langmuir isotherm model assumes that adsorption takes place on a monolayer and there are homogeneous adsorption sites on the surface [26, 27]. The Langmuir isotherm can be expressed by Eqn. 3 (Table 1) where  $V_m$  (mg/g) and  $k$  are the monolayer adsorption capacity and empirical equilibrium constant, respectively. Adsorption on heterogeneous surfaces can be explained by a Freundlich isotherm. In Eqn. 4 the terms  $K_f$  and  $1/n$  denote the adsorption capacity of the adsorbent and adsorption intensity, respectively. The  $n$  value indicates the degree of nonlinearity between solution concentration and adsorption as follows: if  $n=1$ , then adsorption is linear; if  $n<1$ , then adsorption is a chemical process; if  $n>1$ , then adsorption is a physical process. Favorable adsorption conditions can be represented by  $1/n$  values between 0 and 1.

Table 1. Applied isotherm equations

Isotherm	Equation	Isotherm parameters	Plot
Langmuir	$q_e = C_e V_m k / 1 + k C_e$	$k$ and $V_m$	$\frac{C_e}{q_e}$ versus $C_e$ (Eqn. 3)
Freundlich	$q_e = K_f C_e^{1/n}$	$K_f$ and $\frac{1}{n}$	$\log q_e$ versus $\log C_e$ (Eqn. 4)

In this study, the isotherm parameters were determined from the intercept and slope of the concerned plots (Table 1). The Plots of the applied isotherm models have been presented in Fig. 8. The calculated isotherm parameters and  $r^2$  values of the applied isotherms have been summarized in Table 2. As seen from the isotherm studies, the equilibrium data can be best explained by Freundlich isotherms which provide higher  $r^2$  values. These results confirm heterogeneous adsorption surface and multilayer adsorption phenomenon. Furthermore,  $n$  calculated as smaller than 1, then adsorption is a chemical process.



**Figure 8.** Plots of the applied (a) Langmuir (b) Freundlich and Halsey

**Table 2.** Isotherm parameters determined for adsorption of atrazine ( $C_0= 0.2\text{-}2.0$  mg/L, pH= 5.4,  $m=1.2$  g,  $V=50$  ml,  $T= 25$  °C,  $t=120$  min, and  $w=250$  rpm)

Langmuir isotherm constants			Freundlich isotherm constants		
$V_m$	$k$	$r^2$	$K_f$	$n$	$r^2$
23.78	0.098	0.73	2.02	1.24	0.998

## 4 CONCLUSION

Atrazine, used for agricultural purposes, is an important pollutant for the ecosystem. Due to its toxic effect, it is resistant to biological treatment, and it is very difficult to remove by chemical oxidation. Therefore, in this study, a composite adsorbent was produced by using polyaniline and rice husk with a very easy process, and atrazine removal was studied by the adsorption process. The optimum treatment efficiency for removal of atrazine (25 mg / L) was found as 58.3% under 120 min., 5.4 of pH, and 1.2 g / 50 ml, and at this condition, adsorption capacities (qt) were calculated as 0.58 mg/g.

In this study, the maximum pollutants removal was aimed with minimum chemical and energy consumption. For this reason, the natural pH value has been selected as optimum because it provided a similar treatment efficiency with other pH values. On the other hand, at the end of the 120 minutes, further treatment efficiency has not been obtained, so 120 minutes was selected as an optimum. 1.2 g / 50ml has been selected as the optimum adsorbent dosage because it is the point that provides the maximum removal efficiency with a minimum dosage. According to the isotherm studies results, the equilibrium data can be best explained by Freundlich isotherms which provide higher  $r^2$  values (0.998).

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## Note

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## Author Contributions

**Nevin ATALAY GENGEÇ:** Conceptualization, Methodology, Formal analysis, Resources, Writing - Original Draft, Writing - Review & Editing, Visualization, Funding acquisitions  
All authors read and approved the final manuscript.

## Conflict of interest

No conflict of interest was declared by the authors.



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