RESEARCH ARTICLE

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Adsorption of Rhodamine-B from Aqueous Solution Using APTMS and GPTMS Modified Silica Sorbents

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ABSTRACT: In this study, modified silica sorbents were used as sorbents in the adsorption of Rhodamine-B from an aqueous solution. Modified silica sorbents were prepared by sol-gel method using paddy waste ash as a silica source. APTMS ((3-Aminopropyl)trimethoxysilane) and GPTMS ((3-Glycidyloxypropyl)trimethoxysilane) were used as modification agents and prepared samples in this way were denoted as Silica-A and Silica-G, respectively. Prepared sorbents were characterized by FT-IR, SEM, N_2 adsorption-desorption, and zeta potential analyses. The adsorption experiments were conducted under initial pH values between 4 and 12, at 30ºC with a dye concentration of 10 mg/L, 0.05 g sorbent amount (working volume of 50 mL), and 100 rpm shaking rate in a batch system. The highest dye removal percentage was obtained at pH 4 for both sorbents. The dye removal percentage of Silica-A and Silica-G sorbents were found as 17.5 % and 32.1 %, respectively. As the initial pH increased the dye removal percentage decreased. These results demonstrated that GPTMS-modified silica sorbent can be used in the adsorption of Rhodamine-B dye from an aqueous solution.

Keywords: Rhodamine-B, Adsorption, APTMS, GPTMS, Paddy waste ash

INTRODUCTION

The textile, dye, plastic, paper, food, and printing industries widely used synthetic dyes as the colorizing agent. Among them, textile dyeing processes produce a high amount of colorized water (Rai et al., 2005). Colorized wastewater can reduce the light permeability of water and cause decreased photosynthetic activity and increase chemical oxidation needed (COD) for the water (Goscianska et al., 2017). Additionally, the accumulation of dyes in some aquatic organisms causes the formation of undesired toxic and carcinogenic products (Sharma and Uma 2010). Several methods have been applied for the removal of a dye such as electro-oxidation, photodegradation, coagulation, membrane separation, chemical oxidation, and microbial degradation (Sharma et al., 2018). Among these techniques, adsorption is preferred due to its low cost, efficiency, and easy application stages (Liu et al., 2020). In adsorption processes, different sorbent materials (synthetic, natural and agricultural) such as eggshell (Okur, 2013), rice husk ash (Swarnalakshmi et al., 2018), zeolite (Aktı and Okur, 2016), TiO_2, and Rice Husk Ash (Akshaya et al., 2018), KIT-6 (Koyuncu and Okur, 2021a), cellulose aerogel (Dilamian and Noroozi, 2021) have been used.

Recent studies have focused on the use of new aerogel materials in adsorption processes. Aerogels are 3-D interconnected structures obtained by a certain drying process such as supercritical and freeze-drying of the liquid component of the gel (Dilamian and Noroozi, 2021). The aerogel materials with low density, high porosity, and high surface area have been accepted as promising sorbent materials for adsorption studies (Wang et al., 2020). Silica aerogel materials consist of a cross-linked silica network (Maleki et al., 2014). These materials have porous structures (~90%), high surface areas (500-1000 m^2/g), very low densities (~0.03–0.5 g/cm^3), and low thermal conductivities (~0.004-0.03 W/mK) (Li et al., 2020; Owoeye et al., 2020). Due to these properties, it has a field of use in thermal insulation materials, sound insulation, radiation detectors, insulated containers, and refrigerators. Apart from this, silica aerogels are widely used for the separation of oil/water, distillation, adsorption studies, drug release, food industry, building materials, and aerospace industry (Khedkar et al., 2019; Li et al., 2020).

Silica aerogels are generally synthesized by the sol-gel method using alkoxides or organic silicon monomers such as tetraethoxysilane (TEOS), tetramethoxysilane (TMOS), polyethoxydisiloxane (PEDS). However, the storage risks and high costs of these chemicals have led to the shift of studies to green production methods using lower cost and non-toxic starting materials. In this respect, sodium silicate solutions attract attention as a good silica source in industrial and large-scale applications (Owoeye et al., 2020). The synthesis method and the properties of the final material differ according to the silica source used. For example, materials prepared with TMOS, TEOS, and sodium silicate have low density (<0.1 g/cm^3), high optical transmission (>90%), hydrophilic nature, hard and brittle structure. Materials prepared with starting materials such as methyltrimethoxysilane(MTMS), methyltriethoxysilane (MTES) can be opaque, hydrophobic, soft, flexible and have a relatively higher density (>0.1 g/cm^3). (Gurav et al., 2010). The production of silica aerogel by sol-gel method using sodium silicate mainly includes forming a solution (sol) containing Na_2SiO_3, gelation, washing, aging by solvent exchange, surface modification, and drying steps (Li et al., 2020). In the recent studies, it has been seen that biomass residues are used as a green and inexpensive source of silica. It is known that paddy residues have high silica content. When the paddy residues are burned, ash containing about 96% silica is obtained. Silica

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aerogel materials can be produced from sodium silicate obtained by extracting this silica with NaOH (Halim et al., 2020). In this method, first, the silica in the paddy structure is extracted as sodium silicate ($Na_2O.xSiO_2$) with NaOH (Eq.1).

$$xSiO_2 + 2NaOH \rightarrow Na_2O.xSiO_2 + H_2O \quad (1)$$

Then, HCl is added to the medium and the silica network structure that will form the silica aerogel structure begins to form at pH ≤10 (Eq.2).

$$Na_2O.xSiO_2 + 2HCl \rightarrow xSiO_2 + 2NaCl + H_2O \quad (2)$$

Finally, washing with deionized water is applied to separate the Na^+ ions from the aerogel (Feng et al.,2018). The silica aerogel materials are modified with compounds such as Aminopropyltrimethoxysilane (APTMS) and Glycidyloxypropyltrimethoxysilane (GPTMS) to improve some of their properties. The polar OH^- group on the silica surface forms hydrogen bonds by keeping the ambient humidity and causes the material to deteriorate over time (Maleki et al., 2014). By modifying the silica aerogel, a hydrolytically stable hydrophobic aerogel is obtained by replacing the hydrogen in the Si-OH structure with Si-R.

In this study, the adsorption of Rhodamine-B from an aqueous solution was investigated using modified silica sorbents prepared from paddy waste ash as sorbent. Synthesis of modified silica sorbents mainly includes silica extraction from paddy ash with NaOH, gelation at room temperature, aging by solvent exchange, modification, filtration, and freeze-drying steps. In this study, (3-Aminopropyl)trimethoxysilane (APTMS) and (3-Glycidyloxypropyl)trimethoxysilane (GPTMS) were used as modification agents and prepared samples in this way were denoted as Silica-A and Silica-G, respectively. In the adsorption experiments, the effect of initial pH (4-12) and contact time were examined.

MATERIALS AND METHOD

Preparation of Silica Aerogel

In this method, 10 g residual paddy waste ash was treated with 200 mL, 2M NaOH at 100°C for 2 hours to extract the amorphous silica from the ash into the liquid phase. At the end of this stage, the solution was filtered by vacuum filtration and the filtrate was neutralized with 2 M HCl until the pH was 7. At this stage, the filtrate is the sodium silicate solution, and the solid is the carbon-rich material. The gel material formed during the neutralization stage was left to age at room temperature for 20 hours. The material filtered by vacuum filtration was washed with deionized water until the ions in the material were completely removed from the structure. Then, the gel material was placed in an ethanol medium, and solvent exchange was performed every 24 hours for a week. The silica aerogel structure was obtained by drying the material that became alcogel with a solvent exchange. In the study, modified silica materials were prepared by modifying the silica aerogel material with organosilane structures (APTMS and GPTMS) and the prepared samples were freeze-dried under vacuum conditions. The prepared samples in this way were denoted as Silica-A and Silica-G. Figure 1 summarizes the preparation steps of modified silica sorbents.

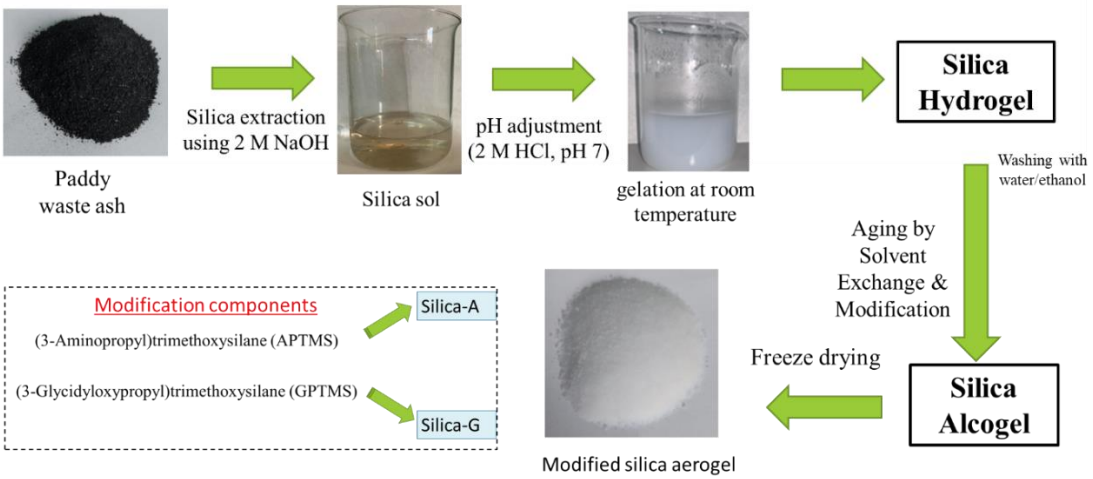

Figure 1. *Synthesis of modified silica sorbent from paddy waste ash*

Characterization of silica aerogel

The structural properties of the aerogels obtained from paddy waste ash and the presence of functional groups were evaluated by FT-IR analysis, morphological properties were evaluated by SEM analysis, pore properties, and surface area were evaluated by N_2 adsorption-desorption analysis. FT-IR analyses were performed in a Jasco 4700 ATR/FT-IR spectrophotometer in the medium infrared region of 4000-400 cm^{-1} with 4 cm^{-1} resolutions. The N_2 adsorption-desorption analyses were conducted using a Quantachrome Autosorb-1C/MS instrument, at 77 K. The surface areas of the sorbents were found by Brunauer-Emmett-Teller (BET) method within the N_2 relative pressure range (P/P^o) of 0.05 - 0.30. Barrett-Joyner-Halenda (BJH) method was applied to obtain pore size distributions of the samples using the desorption values. The zeta potential measurements were carried out for the sorbents to be used in dye removal.

Adsorption Experiments

The modified silica aerogels prepared in this study were used in the Rhodamine B dye adsorption process. In the adsorption experiments, the effect of initial pH (4-12) and contact time were examined at 30°C with a dye concentration of 10 mg/L, 0.05 g sorbent amount (working volume of 50 mL), and 100 rpm in a batch system. The percentage of dye removal and dye removal capacity (q) were calculated with Eq.3 and 4.

$$Dye\ removal\ \% = \frac{(C_o - C_t)}{C_o} * 100 \quad (3)$$

$$q = \frac{(C_o - C_t) * V}{m} \quad (4)$$

In these equations;

C_o : initial dye concentration (mg/L),
C_t : dye concentration in the medium at time t (mg/L),
V : Solution volume (L),
m : amount of sorbent (g),
q : adsorption capacity (mg/g).

RESULTS AND DISCUSSION

In this study, the adsorption of Rhodamine-B from an aqueous solution was investigated using modified silica sorbents prepared from paddy waste ash. It is reported in the literature that paddy waste ash contains a high amount of SiO_2, although it varies according to the place where it is grown (Atta et al., 2012; Aono et al., 2018) As we reported in our previous study (Koyuncu and Okur, 2021b), the paddy waste ash we use contains nearly 85wt% of silica in its structure. This is the main reason to evaluate the paddy waste ash as a silica source.

Modified silica samples were synthesized by using the (3-Aminopropyl)trimethoxysilane (APTMS) and (3-Glycidyloxypropyl)trimethoxysilane (GPTMS) modifying agents. The structural and functional groups present in the sorbent structure were determined by FT-IR analysis. The FT-IR spectrums of the modified samples are given in Figure 2. All the samples exhibited bands around 450 cm^{-1}, 790 cm^{-1} and 1056 cm^{-1}. The peak at 1056 cm^{-1} and accompanying shoulder (about 1200 cm^{-1}) were related to the asymmetric stretching of the Si-O-Si (siloxane) bond of the silica lattice. The bending and symmetrical stretching vibrations of Si-O-Si were observed at 450 cm^{-1} and 790 cm^{-1}wavenumbers, respectively (Merkache et al., 2015). Small peaks observed at 1635 cm^{-1} corresponded to the O-H bending vibrations of water interacting with the surface (Nandiyanto 2016). The characteristics of ATR (Attenuated total reflection) measurements were observed between 1900 and 2400 cm^{-1}as small vibrations. Small peaks that appeared at about 2935 cm^{-1} were related to the C-H stretching vibrations of CH_3 bond of modifying agents (Suddai et al., 2018, Swarna et al., 2018). Stretching vibrations of -NH_2 group of APTMS occurred at 1595 cm^{-1} (Suddai et al., 2018). These results confirm the successful modification of silica samples.

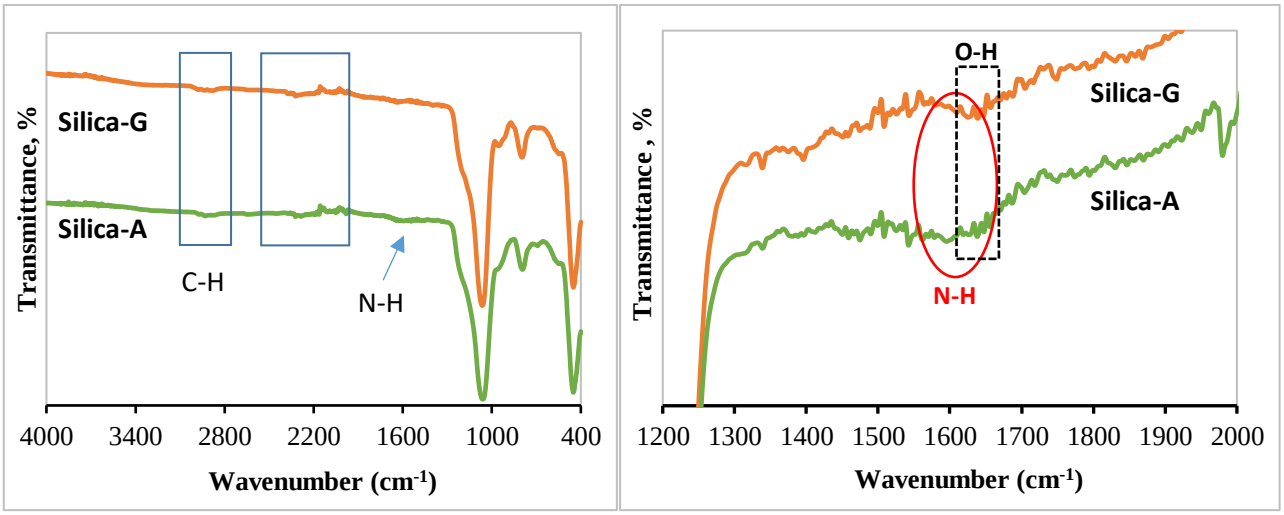

Figure 2. *FT-IR spectrums of the modified-silica sorbents (a) 400-4000 cm^{-1}, (b) 1200-2000 cm^{-1}*

Figure 3 shows the SEM images of modified-silica samples. As reported previously, the spherical particles attracted attention in the SEM images of the unmodified-silica sample (Koyuncu and Okur, 2021b). However, spherical particles were not seen in the modified silica sorbents, and these sorbents exhibited a non-rigid morphological structure.

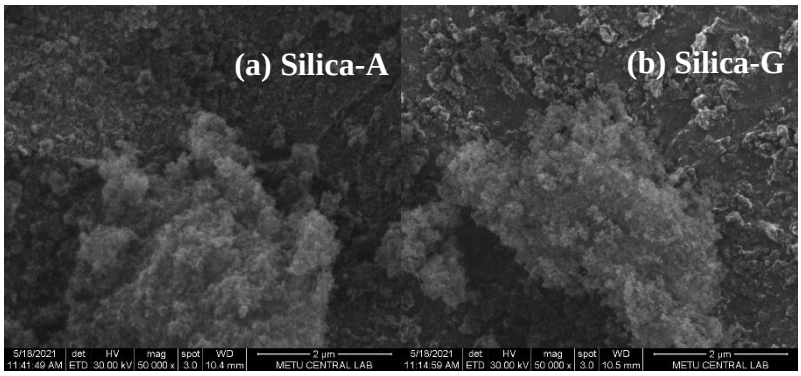

Figure 3. *SEM images of (a) Silica-A and (b) Silica-G sorbents (x 50 000)*

The isoelectric points (pH_{PZC}) of the sorbents were determined by zeta potential curves. Figure 4 shows the surface zeta potential of the Silica-A and Silica-G samples. It was reported in the literature that, the sorbent surface is protonated at pH values lower than pH_{PZC}, and the electrostatic interaction between the anionic dye and the sorbent increases, thus the dye removal percentage increases (Yu et al., 2015). The opposite case is correct for cationic dye. The pH_{PZC} of the Silica-A and Silica-G sorbents were found as 5.2 and 4.2, respectively.

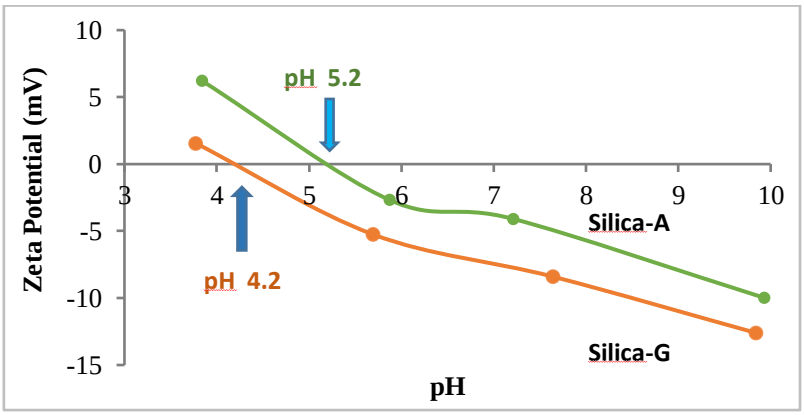

Figure 4. *Zeta potential curves of Silica-A and Silica-G sorbents*

In our previous study, we confirmed that it is possible to prepare a mesoporous unmodified silica sample (with a pore diameter of 17.7 nm) by evaluating the non-porous paddy waste ash as a silica source (Koyuncu and Okur, 2021b). Figure 5 shows the N_2 adsorption-desorption isotherms and BJH pore size distribution curves of modified silica sorbents. The occurrence of pore enlargement after modification can be seen in the BJH pore size distribution curves. The average pore diameters of Silica-A and Silica-G samples were found to be 31.0 and 32.6 nm, respectively. The presence of pores with a diameter greater than 50 nm in Silica-A and Silica-G samples indicated the presence of macropores. As seen in Table 1, modified silica sorbents having high surface area and pore volume can be prepared by using the non-porous paddy waste ash.

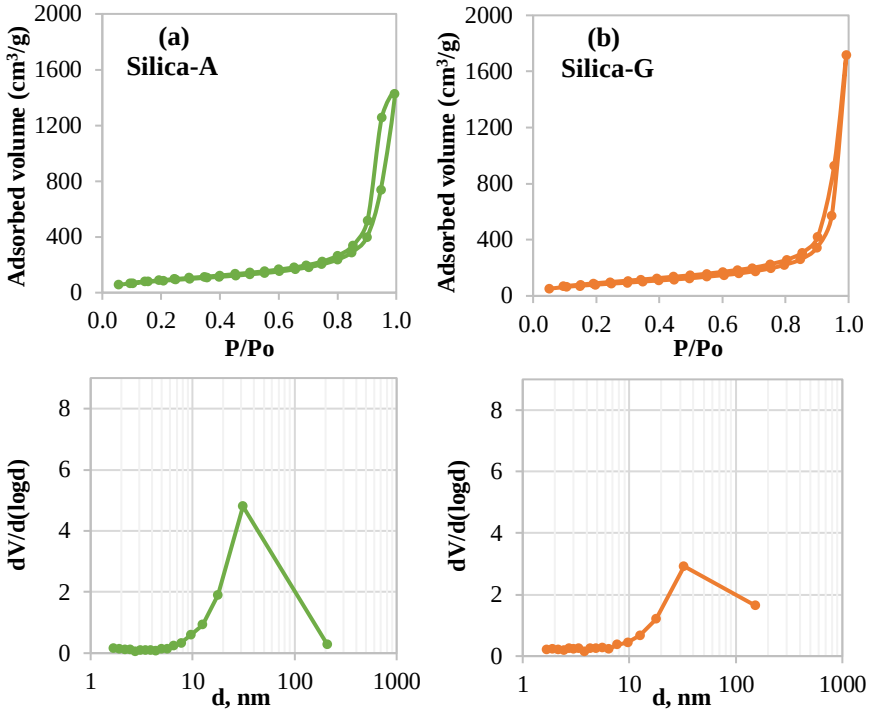

Figure 5. *N_2 adsorption-desorption isotherms and pore size distributions of modified silica samples*

Table 1. *N_2 adsorption-desorption results of modified silica samples*

Sample	Avg. pore diameter, nm	Total pore volume, cm^3/g	Surface area, m^2/g
Silica-A	31.0	2.3	438
Silica-G	32.6	2.7	447

It is known that the pH of the aqueous solution is a very important parameter in dye adsorption. In this study, the adsorption of Rhodamine-B was investigated under different pH values ranging between 4 and 12. In the adsorption experiments, the effect of initial pH (4-12) and contact time were examined at 30 ^oC with a dye concentration of 10 mg/L, 0.05 g sorbent amount (working volume of 50 mL), and 100 rpm in a batch system. Figure 6 shows the dye removal percentage of Silica-A and Silica-G samples. It is seen that the highest dye removal percentage was obtained at pH 4 for both sorbents. The highest dye removal percentage of Silica-A and Silica-G sorbents were found as 17.5 % (at equilibrium time of 10 min) and 32.1 % (at equilibrium time of 20 min), respectively. As the initial pH increased the dye removal percentage decreased.

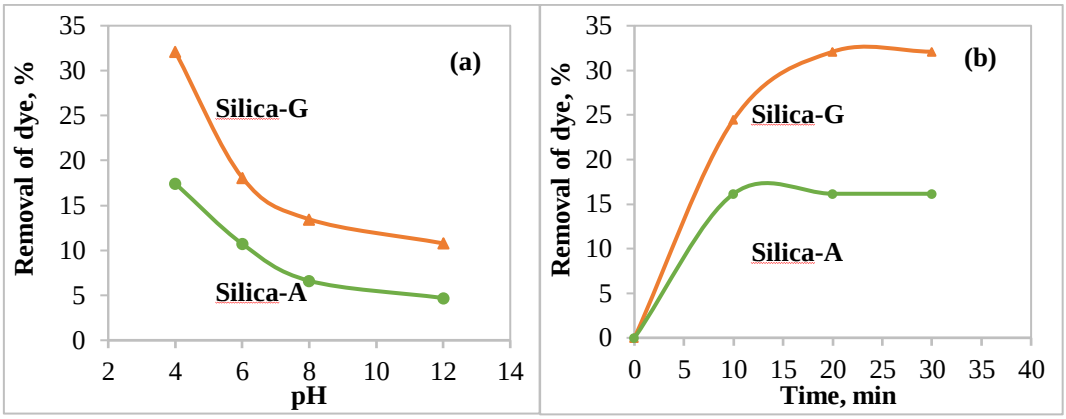

Figure 6. *Adsorption of Rhodamine-B from aqueous solution (a) the effect of pH (b) the effect of contact time (30ºC, with a dye concentration of 10 mg/L, 0.05 g sorbent, 50 mL solution)*

Table 2 includes some adsorption results of different silica-based sorbents and the results obtained in this study. These results indicated that the GPTMS modified silica sorbent had a better ability to adsorb Rhodamine-B dye from an aqueous solution.

Table 2. *Adsorption results of different silica-based sorbents reported in the literature and sorbents used in this study*

Sorbent	Dye	q (mg/g)	References
Cationic-modified silica gel	Reactive Black 5	190.00	Zhang et al., 2019
	Reactive Red 239	178.20	
Si-APTES-BP	RB19	37.45	Banaei et al., 2017
	RY84	32.36	
Monoamine modified silica particles	Acid Orange 12	15.50	Donia et al., 2009
	Acid Orange 10	58.10	
Magnetic silica immobilized *P.fluorescens*	Rhodamine B	229.6	Joshiba et al., 2021
N-halamine modified mesoporous silica	Rhodamine B	31.97	Wang et al., 2021
PCS	RTGN	26.00	Koyuncu and Okur, 2021b
PS		1.40	
PA		1.95	
Silica-A	Rhodamine B	6.24	This study
Silica-G		12.38	

CONCLUSION

In this study, the adsorption of Rhodamine-B from an aqueous solution was investigated using APTMS and GPTMS modified silica sorbents. Modified silica sorbents were prepared by using the paddy waste ash as a silica source. The characterization results indicated that it is possible to prepare porous modified silica samples with high surface area and pore volume from non-porous paddy waste ash. The FT-IR results confirmed that the modification was done successfully. Both sorbents exhibited the highest removal percentage at pH 4 conditions. GPTMS modified silica sorbent (Silica-G) showed a better ability to adsorb Rhodamine-B dye from an aqueous solution.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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