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Original Research

Rubber Foam Incorporating Activated Carbon and TiO_2 for the Adsorptive Removal of Rice Starch Wastewater

Ratana Sananmuang[1, 2], Wikorn Punyain[1], Duangdao Channei[1]°*

[1]Department of Chemistry, Faculty of Science, Naresuan University, Phitsanulok, 65000, Thailand
[2]Center of Academic Excellence for Petroleum, Petrochemical, and Advanced Materials,
Naresuan University, Phitsanulok, 65000, Thailand

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Abstract

White rice starch is a major pollutant in wastewater from rice noodle production. This study developed a composite adsorbent (RFAcTD) by incorporating activated carbon (Ac) and TiO_2 (TD) into rubber foam (RF) at varying Ac:TD ratios (4:1, 2.5:2.5, and 1:4). Adsorption behavior followed the Langmuir isotherm for RFAcTD (4:1 and 2.5:2.5), while the TiO_2-rich RFAcTD (1:4) fit the Freundlich model, indicating multilayer adsorption. Thermodynamic analysis via the Van't Hoff plot yielded a ΔH of -83.82 kJ/mol, confirming an exothermic adsorption mechanism driven by strong hydrogen bonding between starch hydroxyl groups and surface functionalities on the activated carbon and TiO_2 of the RFAcTD adsorbent. DFT calculations confirmed that rice starch forms a stable complex with the modified rubber (RFAcTD) through hydrogen bonding, with interaction energies of -97.72 kJ/mol in the gas phase and -71.86 kJ/mol in the aqueous phase. Application to real wastewater from Thai rice noodle factories demonstrated effective starch removal, supporting the potential of RFAcTD for practical wastewater treatment.

Keywords: rubber foam, adsorption, rice starch, activated carbon, titanium dioxide

Introduction

Wastewater discharge from food processing industries has emerged as an increasingly critical environmental concern. It is especially notable in small food factories, such as Thai fermented rice vermicelli noodle (Khanom Jeen) factories and rice noodle (Lod Chong) factories, which are considered types of food factories that have both small and large scales of production, and are located throughout all regions of Thailand. The wastewater generated from Thai fermented rice vermicelli production contains rice starch from soaking and washing processes, which is often discharged into public water bodies, leading to fermentation and unpleasant odors. Various treatment methods have been studied, including anaerobic digestion [1] and reverse osmosis after sedimentation and microfiltration [2]. Adsorption is widely recognized for its simplicity and effectiveness. Some researchers have explored valorization approaches such as microalgae

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*e-mail: duangdaoc@nu.ac.th
°ORCID iD: 0000-0001-8951-3934

cultivation [3], bioflocculant production [4], and ethanol synthesis [5]. However, few studies have focused on starch removal via adsorption. Thus, developing low-cost, sustainable adsorbents with enhanced surface area, porosity, and chemical affinity is essential for efficient starch removal. Activated carbon prepared from *Mimosa Pigra* L. is an interesting material because this plant is an invasive weed widespread in the wetlands of northern Thailand. According to Singhasiri et al. (2014) [6], *Mimosa pigra* L. serves as a promising precursor for activated carbon production, producing material with a high iodine number, ranging from 631 to 966 mg/g. If the treatment relies solely on adsorption, pollutant molecules are confined to the surface of the adsorbent rather than being transformed or eliminated. Therefore, subsequent photocatalysis under light irradiation is employed to degrade the adsorbed molecules. The incorporation of titanium dioxide (TiO_2), a benchmark photocatalyst, into the rubber foam matrix not only facilitates the photoactive phase but also enhances the mechanical stability and adsorption efficiency of the composite adsorbent. As reported by Balasubramanian et al. (2019) and Xiong et al. (2019) [7, 8], TiO_2 serves as a reinforcement and physical cross-linking agent in polymeric and polysaccharide-based systems, contributing to enhanced structural integrity, porosity, and surface functionality. The integration of rubber foam with activated carbon derived from *Mimosa pigra* L. and TiO_2 presents distinct advantages over conventional powdered adsorbents. Rubber foam provides a lightweight, porous, and elastic structure that not only facilitates the dispersion of active components but also enhances mechanical stability during operation. This system differs from previous TiO_2/activated carbon composites or polymeric foams by its lightweight, floating nature, which allows easy recovery after treatment. Utilizing low-cost natural rubber available in Thailand, the material adds value to local resources while maintaining simple preparation. Unlike fine powdered adsorbents, which are difficult to separate from treated water and may require additional filtration steps, the rubber foam–based composite can be easily removed from the solution after adsorption, reducing operational complexity and minimizing secondary waste. The incorporation of activated carbon from *Mimosa pigra* L. contributes a high surface area and abundant functional groups for starch binding, while TiO_2 enhances structural integrity, surface hydrophilicity, and interaction with starch molecules. This integrated design ensures a reusable, easily handled, and efficient adsorbent suitable for practical wastewater treatment applications. Thus, the aim of this study was to develop a rubber foam–based adsorbent incorporating activated carbon derived from *Mimosa pigra* L. and TiO_2. The study included characterization of the adsorbent morphology, as well as evaluation of its performance in removing starch from synthetic starch-containing wastewater. Additionally, the interaction between the adsorbent and adsorbate was investigated using Density Functional Theory (DFT) calculations.

Materials and Methods

Starch and natural rubber dispersion were procured from Loba Chemie Pvt. Ltd. (India) and Lucky Four Co., Ltd. (Thailand), respectively. All other analytical reagent (A.R.) grade chemicals were obtained from Merck (Germany).

Adsorbent Preparation

A total of 100 g of 60% natural rubber dispersion was blended with polymer additives consisting of 1.5 g of 10% potassium oleate, 2.0 g of 50% sulfur dispersion, 1.0 g of 50% zinc diethyldithiocarbamate dispersion, 1.0 g of 50% zinc mercaptobenzothiazole dispersion, 5.0 g of 50% zinc oxide dispersion, and 0.7 g of 33% diphenylguanidine. The mixture was stirred until a homogeneous dispersion was obtained. Activated carbon (Ac) and TiO_2 powder were then incorporated into the mixture at five Ac:TiO_2 ratios (4:1, 1:4, and 2.5:2.5 w/w), producing the composite rubber foam adsorbents (RFAcTD). The resulting mixtures were poured into square trays and cured in an oven at 100ºC for 2 h. The prepared RFAcTD samples were stored for further use. Morphological characterization was carried out using scanning electron microscopy (SEM) with a Thermo Scientific Apreo instrument.

Adsorption Studies

The adsorption efficiency for starch removal from synthetic starch wastewater was evaluated through batch experiments. The influence of initial starch concentration (100, 300, 500, 1000, and 2000 mg/L) and temperature (30ºC, 35ºC, and 40ºC) was systematically investigated.

The Chemical Oxygen Demand (COD) test was carried out following the standard procedure recommended by APHA, AWWA, and WEF (1998) using the potassium dichromate ($K_2Cr_2O_7$) digestion method. In this method, a known volume of sample was transferred into a reflux flask and mixed with a standard $K_2Cr_2O_7$ solution of known normality in a strongly acidic medium. The mixture is then digested at 150ºC for 2 h, allowing the dichromate to oxidize the organic matter present in the sample. After cooling to room temperature, the remaining unreacted dichromate is titrated with a standard ferrous ammonium sulfate (FAS) solution using ferroin as an indicator. A blank, prepared under identical conditions with distilled water, is titrated in parallel. The difference in FAS volume between the blank and the sample represents the dichromate consumed in oxidizing the organics. The result represents the COD, expressed in mg O_2/L, indicating the total quantity of oxygen required to chemically oxidize the organic matter in the sample. In the case of real wastewater, the sample may contain a complex mixture of organic compounds such as proteins, fats, sugars, dyes, surfactants, and various other oxidizable substances. Consequently, the COD value represents the total oxygen demand required to oxidize all organic matter present, not just carbohydrates.

Firstly, the RFAcTD samples were randomly cut into small pieces, and 0.10 g was weighed and mixed with 20 mL of starch solution. The solution pH was adjusted to the predetermined optimum value using 1.0 M NaOH or 1.0 M HNO_3. The mixture was stirred at 300 rpm and 30ºC for a total of 10 h to determine the equilibrium time. The results indicated that maximum adsorption was achieved at 4 h, after which the adsorption remained constant. The RFAcTD was subsequently separated from the starch solution by filtration through No. 5 filter paper, and the residual starch concentration in the filtrate was determined using the COD method described above. The procedure was repeated at elevated temperatures of 35ºC and 40ºC under otherwise identical experimental conditions.

In addition, the photocatalytic activity of the synthesized rubber foam incorporated with activated carbon and titanium dioxide (RFAcTD) was evaluated against a high concentration of rice starch in aqueous solution. Typically, 0.01 g of RFAcTD was added to a beaker containing 50 mL of rice starch solution with an initial concentration (C_0) of 2000 mg/L, corresponding to another unit equivalent of 10,000 mg/g. The suspension was then irradiated with UV light in the wavelength range of 200-400 nm (120 W; eight lamps, each rated at 15 W). The amount of starch adsorbed onto RFAcTD at a given time was calculated as follows:

$$q_t = \frac{(C_0 - C_t) \times V}{W} \tag{1}$$

where q_t represents the amount of starch adsorbed (mg/g), C_o and C_t (mg/L) denote the initial and time-dependent starch concentrations, respectively, V (L) is the solution volume, and W (g) is the mass of RFAcTD used [9].

Adsorption Isotherms

Adsorption isotherms are essential models for determining adsorption parameters, as they describe the interaction between adsorbate molecules in the liquid phase and the surface of the solid adsorbent [10]. In this study, the Langmuir and the Freundlich isotherm models were applied to evaluate the influence of temperature on the starch adsorption capacity of the synthesized RFAcTD. The Langmuir isotherm, a linear model frequently used in adsorption studies, assumes monolayer adsorption onto a homogeneous adsorbent surface with a finite number of identical adsorption sites and negligible intermolecular interactions [11]. The linearized form of the Langmuir Equation is expressed as:

$$\frac{C_e}{q_e} = \frac{1}{X_m K_L} - \left(\frac{1}{X_m}\right) C_e \tag{2}$$

where K_L is the Langmuir adsorption constant (L/mg), x_m is the maximum adsorption capacity (mg/g), q_e is the amount of adsorbate adsorbed (mg/g), and C_e is the equilibrium adsorbate concentration (mg/L). From the linear plot of C_e/q_e versus C_e, the parameters x_m and K_L are determined from the slope and intercept, respectively [11, 12].

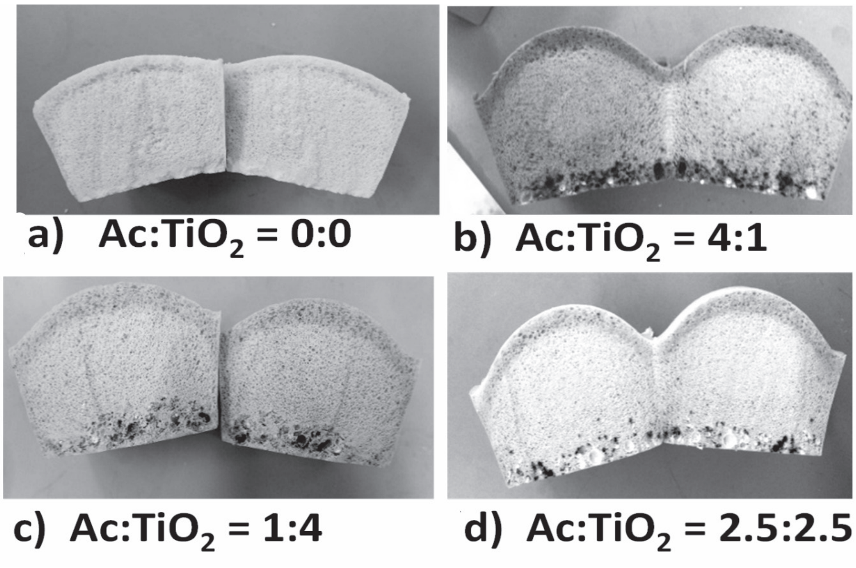
a) Ac:TiO_2 = 0:0
b) Ac:TiO_2 = 4:1
c) Ac:TiO_2 = 1:4
d) Ac:TiO_2 = 2.5:2.5

Fig. 1. Physical characteristics of rubber foams incorporated with activated carbon and titanium dioxide (RFAcTD) at different ratios of activated carbon to TiO_2.

The Freundlich isotherm is an empirical model used to describe adsorption on heterogeneous surfaces with non-uniform distribution of adsorption energies [13, 14]. The linearized form of the Freundlich Equation is expressed as:

$$\log q_e = \log K_F + \left(\frac{1}{n}\right)\log C_e \qquad (3) \tag{3}$$

where K_F is the Freundlich adsorption constant (mg/g), and n denotes the adsorption intensity or a measure of surface heterogeneity. The slope (1/n) and intercept (K_F) are determined from the linear plot of log q_e versus log C_e

Adsorption Thermodynamics

Thermodynamic parameters, such as enthalpy (ΔH), offer valuable insight into the energetic changes accompanying the adsorption process. These parameters were calculated from the Langmuir equilibrium constant using the Van't Hoff Equations as follows:

$$\ln K_L = \frac{\Delta S}{R} - \frac{\Delta H}{R}\left(\frac{1}{T}\right) \tag{4}$$

where K_L is the Langmuir equilibrium constant (L/mg), R is the universal gas constant (8.314 J/mol.K), and T is the absolute temperature (K) [15, 16]. The value of ΔH was obtained from the slope of the Van't Hoff plot of ln K_L versus 1/T.

Results and Discussion

Characterization of RFAcTD Adsorbents at Different Activated Carbon/TiO_2 Ratios

The physical characteristics of synthetic rubber foams (RF) prepared with varying ratios of activated carbon (Ac) and TiO_2 (TD) are shown in Fig. 1. All RFAcTD samples exhibited a porous structure with numerous visible holes. During oven curing of the rubber for approximately 2 h, the activated carbon particles may have settled toward the bottom of the mold, resulting in the presence of black particles in the RFAcTD samples containing activated carbon. The abundant porous structure observed in these prepared foams is expected to enhance their adsorption performance. The morphology of RFAcTD, shown in Fig. 2(a-d), exhibits similar surface characteristics across all samples. Numerous air holes, generated during the foaming process, are clearly visible and vary in size throughout the structure. The samples display a gray-black coloration with uniformly dispersed white spots attributed to TiO_2 particles.

Adsorption Studies

Equilibrium adsorption was evaluated for RFAcTD (4:1) as a representative formulation over 1-10 h at an initial starch concentration of 100 mg/L. The adsorption capacity increased monotonically with contact time and reached equilibrium at ~4 h (Fig. 3a)). Accordingly, the adsorption capacities (q_e) obtained at 4 h were used for subsequent isotherm analyses.

Adsorption isotherms were assessed to elucidate the underlying mechanisms, with all RFAcTD formulations evaluated at 30ºC using equilibrium data obtained across starch concentrations of 100, 300, 500, 1000, and 2000 mg/L. The Langmuir model showed the best fit for RFAcTD (4:1) and RFAcTD (2.5:2.5), as evidenced by their higher R^2 values, suggesting monolayer adsorption on a homogeneous surface (Fig. 3b)) [17, 18]. In the Langmuir plot section, a comparison of the Langmuir parameters (X_m and K_L) for the 2 samples (4:1 and 2.5:2.5) revealed that RFAcTD (4:1) exhibited a higher maximum adsorption capacity (X_m = 22.37 mg/g) than the 2.5:2.5 sample, as summarized in Table 1. For RFAcTD (1:4), the adsorption data conformed more

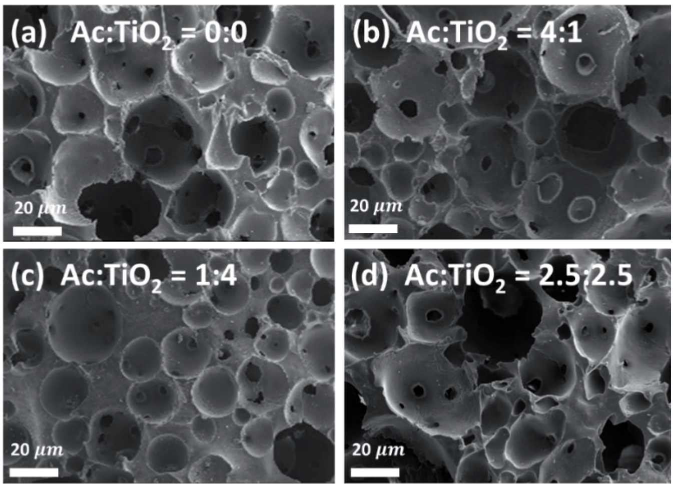

Fig. 2. SEM micrographs of rubber foams incorporated with Ac and TiO_2 at different ratios: a) 0:0, b) 4:1, c) 1:4, and d) 2.5:2.5.

Table 1. Adsorption isotherm model parameters for all adsorbents at 30ºC.

RFAcTD (Ac:TD)	Langmuir Isotherm			Freundlich Isotherm		
	X_m (mg /g)	K_L (L/mg)	R^2	1/n	K_f (mL/mg)	R^2
(4:1)	22.37	0.037	0.9753	0.3689	1.991	0.7950
(2.5:2.5)	21.55	0.007	0.9971	0.3824	1.098	0.9237
(1:4)	54.05	0.003	0.8833	0.5193	1.421	0.9886

closely to the Freundlich model (Fig. 3c)), implying that the higher TiO_2 content enhanced multilayer adsorption of starch molecules onto TiO_2 surfaces, in agreement with the observations reported by Anaya-Esparza et al. (2020) [19].

Based on the isotherm parameters in Table 1, RFAcTD (2.5:2.5) was selected as the representative adsorbent for photocatalytic assessment under UV irradiation at a starch concentration of 2000 mg/L. In this part, the RFAc matrix acts as the adsorptive medium, while TiO_2 provides the photocatalytic functionality; the enhanced performance is presented in Table 2. The adsorption-only process achieved a maximum removal efficiency of 74.67%. This performance, without UV irradiation, exceeds that reported by Chen et al. [20], who obtained an adsorption efficiency of approximately 48% for potato starch wastewater using a carbon adsorption method under optimal conditions (activated carbon particle size of 40 mesh, dosage of 5 g, adsorption time of 1 h, temperature of 27ºC, and pH 5). These results indicate that adsorption efficiency strongly depends on several parameters, including the adsorbent type, wastewater composition, and operating conditions.

Coupling adsorption with photocatalysis substantially improved performance, achieving a removal capacity of 81.69%. This demonstrates that the synergistic adsorption–photocatalysis process contributes to a higher removal performance than adsorption alone, likely due to the photodegradation of starch molecules adsorbed on the surface coupled

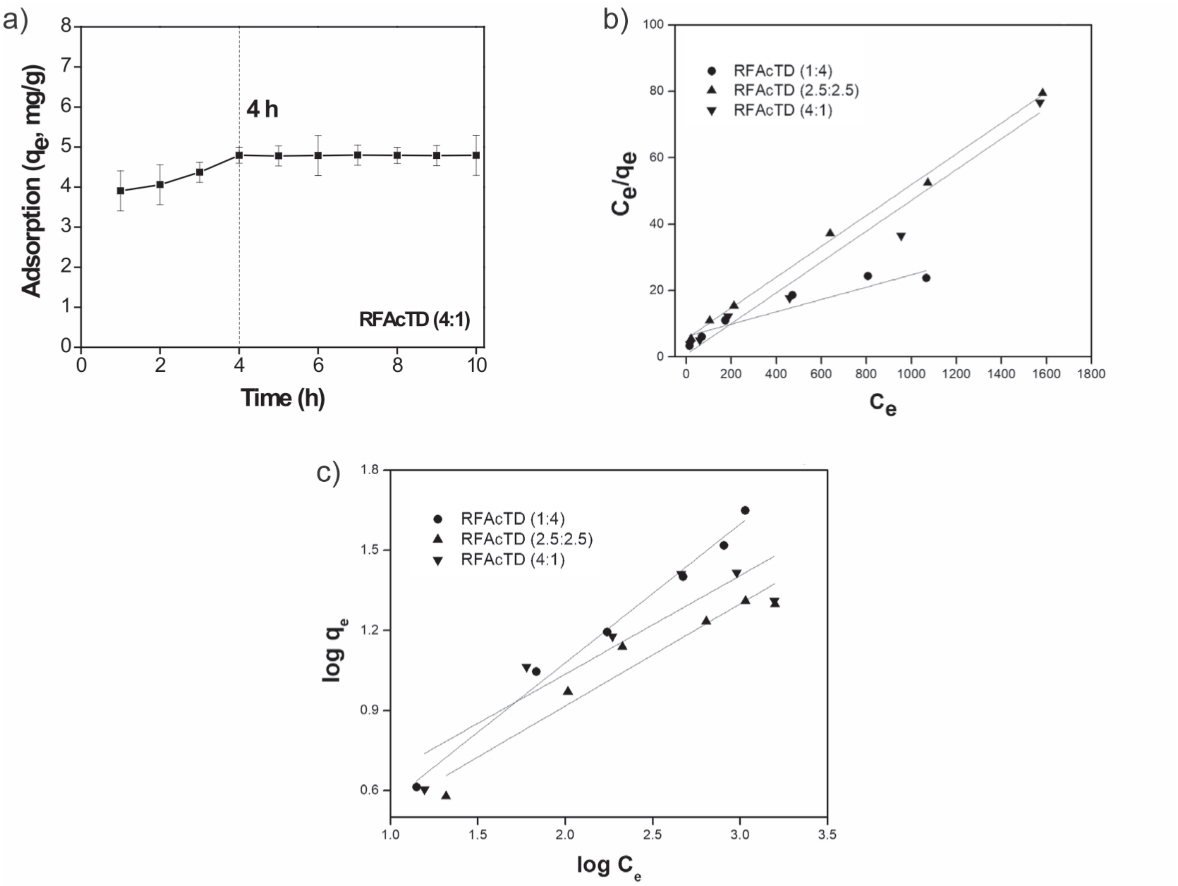

Fig. 3. a) Equilibrium study for RFAcTD (4:1), b) Langmuir linearized plot, and c) Freundlich linearized plot.

Table 2. Starch removal performance of the selected RFAcTD (2.5:2.5) adsorbent under UV irradiation in the presence of TiO_2 photocatalyst.

Adsorbent	Starch Concentration	Adsorption Process		Adsorption process combined with Photocatalysis	
		Remaining Starch	Adsorption Efficiency	Remaining Starch	Removal Efficiency
RFAcTD (2.5:2.5)	2000 mg/L	2,533 mg/g	74.67%	1,831 mg/g	81.69%
	10,000 mg/g				

Note: The solution volume was 50 mL, and the mass of RFAcTD used was 0.01 g.

with the generation of reactive oxidative species under irradiation. Fig. 4 illustrates the combined oxidation and reduction processes that drive the efficient photocatalytic mineralization of starch in aqueous systems. When TiO_2 is irradiated with UV light, photons with energy equal to or greater than its bandgap excite electrons (e^-) from the valence band (VB) to the conduction band (CB), generating electron–hole pairs. The electrons in the CB reduce dissolved oxygen (O_2) to superoxide radicals ($•O_2^-$), which decompose the adsorbed rice starch into smaller intermediates. Simultaneously, holes (h^+) in the VB oxidize water or surface hydroxyl groups to produce hydroxyl radicals (•OH), highly reactive species that further mineralize starch molecules into CO_2, H_2O, and other small by-products.

Subsequently, RFAcTD (2.5:2.5) was employed for practical application studies using real wastewater samples. Starch removal was investigated in three types of wastewaters derived from small fermented rice noodle factories in Sukhothai Province: soaking water from fermented Thai rice vermicelli noodles (S1), washing water from boiled Thai rice vermicelli noodles (S2), and soaking water from mixed flour used for rice noodle preparation (S3). As summarized in Table 3, starch removal efficiencies for S1-S3 ranged from 13.33% to 28.33%, indicating rather low adsorption performance. This could be attributed to the compositional complexity of Thai rice vermicelli and rice noodle doughs, which typically contain multiple flour types such as rice flour, mung bean flour, and tapioca flour.

These starches differ in molecular size, morphology, and protein content, which may influence their interaction with RFAcTD. As reported by Tonsuk et al. (2025) [21], mung bean starch typically forms spherical particles, whereas rice starch granules are polygonal and incorporate variable levels of protein. Such differences in particle characteristics likely influence the adsorption behavior observed in this study.

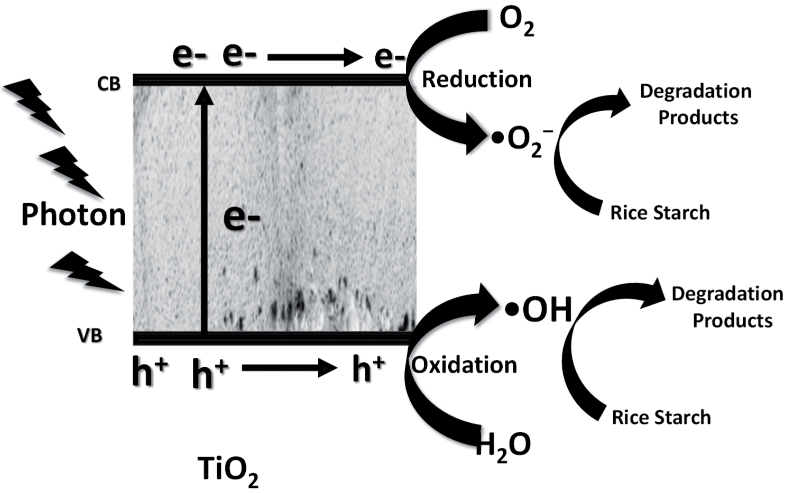

Fig. 4. Proposed photocatalytic degradation mechanism of rice starch on TiO_2 under UV irradiation.

Table 3. Adsorption performance of RFAcTD (2.5:2.5) for starch removal from real wastewater samples.

Adsorbent	Wastewater Sample		
	S1 (mg/g)	S2 (mg/g)	S3 (mg/g)
RFAcTD (2.5:2.5)	13.33%	26.00%	28.33%

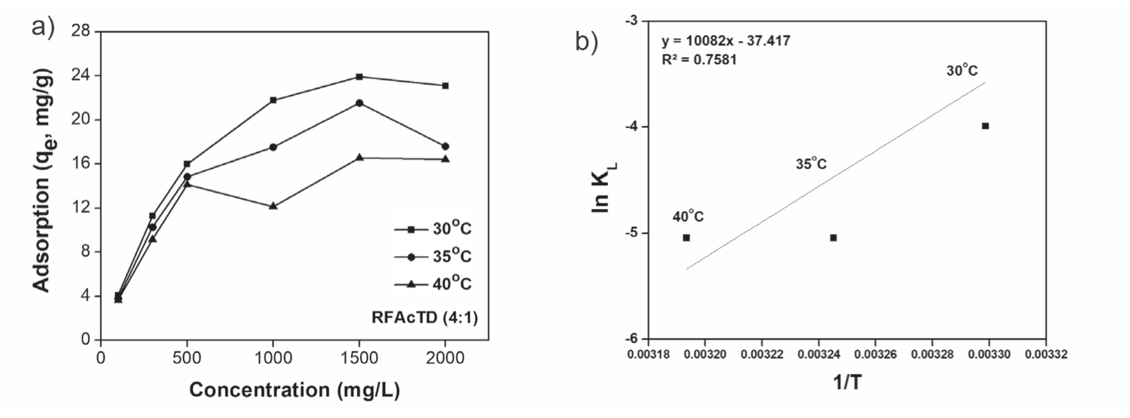

Fig. 5. a) Effect of temperature on adsorption capacity and b) Van't Hoff plot for determination of enthalpy change.

To investigate the effect of temperature on adsorption capacity, the adsorption behavior of RFAcTD (4:1) was examined at starch concentrations of 100, 300, 500, 1000, 1500, and 2000 mg/L under three temperature conditions (30ºC, 35ºC, and 40ºC), as illustrated in Fig. 5a). The observed reduction in adsorption capacity (q_e) at elevated temperatures suggests that the process is exothermic, involving heat release following adsorbate–adsorbent interactions. The enthalpy change (ΔH) for the adsorption process was calculated from the Van't Hoff plot of ln K_L versus the reciprocal of temperature (30, 35, and 40ºC), using equilibrium data at starch concentrations of 100, 300, 500, and 1000 mg/L, as shown in Fig. 5b). A calculated ΔH of approximately -83.82 kJ/mol (from the slope = -ΔH/R) verifies that the adsorption process is exothermic. The predominant adsorption mechanism is attributed to strong hydrogen-bonding interactions between the hydroxyl (–OH) groups of starch molecules and the surface functional groups on the activated carbon and TiO_2 components of the RFAcTD adsorbent [22, 23].

Density Functional Theory (DFT) Investigations

For a more advanced investigation, this study performed calculations to confirm the hydrogen bonding between rice starch and the modified rubber. Rice starch primarily comprises amylose and amylopectin, forming a large branched polymer with thousands of glucose units. In this work, full Density Functional Theory (DFT) calculations were carried out using small oligosaccharides, such as maltose or maltotriose, as simplified molecular models to represent the repeating glucose units, while acetic acid (CH_3–COOH) was used to represent the modified rubber containing carboxylic groups (–COOH). The results demonstrated that the carboxylic groups (–COOH) on the modified rubber surface can interact with the hydroxyl groups (–OH) of rice starch. The three-dimensional geometries of maltotriose, acetic acid, and the resulting maltotriose–carboxylic complex were constructed using the Gabedit software package [24]. The geometry optimization and frequency calculation were done at the B3LYP-D3/cc-pVDZ level of theory, including the effect of short-range dispersion interaction, yielding an optimized geometry located on the local minimum of the potential energy surface (PES). DFT calculations were performed using the ORCA program package [25]. The optimized molecular structures of maltotriose, acetic acid, and the maltotriose–carboxylic complex at the B3LYP-D3/cc-pVDZ level in the aqueous phase are shown in Fig. 6.

It was found that the calculated intermolecular hydrogen bond distances between hydrogen and oxygen atoms in the maltotriose–carboxylic complex were

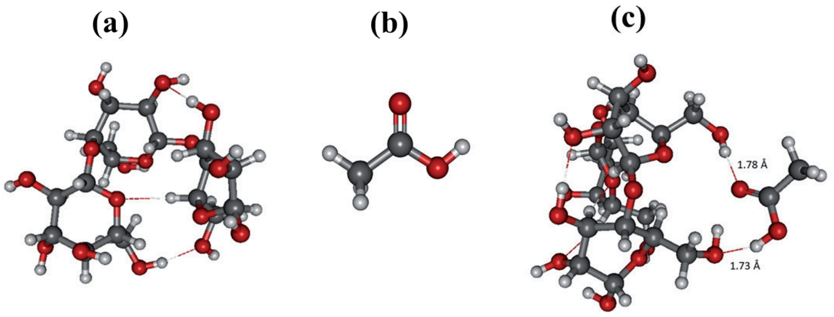

Fig. 6. a) Optimized molecular structure of maltotriose, b) acetic acid (representing the modified rubber), and c) maltotriose–carboxylic complex optimized at the B3LYP-D3/cc-pVDZ level in aqueous phase.

Table 4. Calculated interaction energy (ΔE) of the maltotriose–carboxylic complex in both gas and aqueous phases at the B3LYP-D3/cc-pVDZ level.

	DE (kJ/mol)	
	Gas phase	Water solvent
Maltotriose–Carboxylic	−97.72	−71.86

1.78 Å and 1.73 Å, respectively, as shown in Fig. 6. The interaction energy (ΔE) of the maltotriose–carboxylic complex in both gas and aqueous phases was calculated using the optimized geometries at the B3LYP-D3/cc-pVDZ level, as expressed in Equation (5), and the results are summarized in Table 4.

$$\Delta E = E_{AB} - E_A - E_B \tag{5}$$

Where E_A is the energy of the subsystem maltotriose, E_B is the energy of the subsystem acetic, E_{AB} is the energy of the maltotriose–carboxylic complex.

The calculated interaction energies of the maltotriose–carboxylic complex in the gas and aqueous phases were -97.72 kJ/mol and -71.86 Kj/mol, respectively. The solvation effect in water reduced the interaction energy by approximately 30 Kj/mol compared to that in the gas phase. These negative interaction energies indicate that maltotriose forms a stable complex with acetic acid (representing the modified rubber) in both phases. The DFT results therefore confirm that the modified rubber can interact with rice starch through hydrogen bonding.

Conclusions

Composite rubber foam adsorbents incorporating activated carbon and TiO_2 (RFAcTD) demonstrated strong potential for the removal of rice starch from aqueous solutions and real wastewater streams. The material's porosity and abundance of surface functional groups enabled effective adsorption, particularly for the 4:1 and 2.5:2.5 Ac:TiO_2 ratios. Adsorption behavior was predominantly monolayer in nature, following the Langmuir model, while a higher TiO_2 content promoted multilayer uptake consistent with the Freundlich model. Thermodynamic analysis based on the Van't Hoff equation confirmed that the adsorption process was exothermic, involving heat release and primarily governed by hydrogen-bonding interactions, with an enthalpy change of -83.82 kJ/mol. This finding was further supported by DFT calculations, which revealed the formation of a stable complex. Application to wastewater from Thai rice noodle production further validated the practical utility of RFAcTD for treating starch-rich effluents, offering a promising alternative method for wastewater remediation.

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Conflict of Interest

The authors declare no conflict of interest.

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