

Fenton-like modification of Bauhinia tomentosa seedpod for improved sequestration of methylene blue from water

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Research Article

Keywords: Adsorption, emerging contaminants, methylene blue, Fenton-like modification, Bauhinia tomentosa

Posted Date: January 22nd, 2025

DOI: <https://doi.org/10.21203/rs.3.rs-5856675/v1>

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Additional Declarations: The authors declare no competing interests.

Abstract

Water pollution by dyes remain an important problem that constantly reduces water quality, creating hazard to aquatic flora and fauna, as well as terrestrial life. Although adsorption has been a favourite technique exploited to remediate this growing menace due to its cost-effectiveness and environmental friendliness, adsorbents remain limited in their capacity to efficiently remove emerging dye contaminants from wastewater. In this work, we present an efficient method to improve the sequestration of an emerging contaminant, methylene blue dye (MB), from water through Fenton-like modification of adsorbents. Low-cost and readily-available *Bauhinia tomentosa* seedpod (BTSP) was modified with recyclable FeOCl nanosheets to produce Fenton-BTSP adsorbents. The modified and unmodified adsorbents were characterized using Fourier-transform infrared spectroscopy, dynamic light scattering, energy dispersive X-Ray, and scanning electron microscopy. Results showed successful modification of BTSP to form fine agglomerated particles with enhanced surface areas and pore spaces. Adsorption studies revealed optimal adsorption at pH 10, with equilibrium reached in 30 mins for the adsorbents. Isotherm modelling of the adsorption data suggests formation of multiple layers of MB molecules on the adsorbents at maximum monolayer capacity of 190.63 mg/g and 520.83 mg/g for BTSP and Fenton-BTSP, respectively. Kinetics modelling revealed chemical interaction between the adsorbents and dye molecules at a rate that is higher for Fenton-BTSP. Adsorbent regeneration was also improved after Fenton-like modification, contributing to the potential of Fenton-BTSP for industrial applications.

1. Introduction

The consistent release of toxic effluents from industrial units to water bodies poses greatest risk to the quality of water, the largest irreplaceable asset given to man. Low water quality is a serious problem to aquatic flora and fauna, and has made access to portable water by terrestrial organisms almost impossible in developing and underdeveloped nations (Sun and Lockaby, 2012). To curtail this, the United Nations as part of its sustainable development goals (SDGs), has become more intentional in ensuring that portable water is available to all and sundry by 2030 (UN, 2015).

Effluents from industries contain undesirable and potentially toxic chemicals in the form of textile dyes, pharmaceuticals, agrochemicals, among others. The components of these chemicals compromise the integrity and portability of water and render it unsafe for use (Caldwell et al., 2012). According to the World Health Organization (WHO) report in 2012 (WHO, 2012), about two million people die from diseases that arise from water pollution. In Nigeria, poor access to clean and portable water is a serious menace associated with the high rate of morbidity and mortality especially in infants and children. In a study carried out by the United Nations International Children's Emergency Fund (UNICEF), over seventy thousand children below the age of five die yearly as a result of water-borne diseases (UNICEF, 2014).

Dyes is one of the major sources of water pollution. Dyes are used in almost every aspect of human life, particularly in the textile, leather, paper, rubber, plastics, printing, cosmetics, food, and pharmaceutical industry. Textile dyeing is a well-known and complicated manufacturing process that releases over 140,000 tons of untreated organic dyes into water annually worldwide (Ren et al., 2021). Dyes are generally resistant to light and oxidizing agents, and over ten thousand different dyes are commercially available, which are used to produce million tons of dyes yearly, majority of which are carcinogenic and non-degradable. This makes dyes the most abundant and arguably the most dangerous water pollutants (Robinson et al., 2001; Golka et al., 2004).

Several water remediation methods have been explored and several attempts have been made to eliminate the menace posed by dyes on water. Coagulation (Hanif et al., 2021; Garvasis et al., 2020), chemical oxidation (Chan et al., 2012), photocatalysis and electrocatalysis (Yang et al., 2021; Chem et al., 2020; Zhang et al., 2020), membrane filtration (Sz et al., 2020; Schwayze et al., 2020), dialysis, ion exchange (Wang et al., 2014), solvent extraction, reverse osmosis, and adsorption (Tseng et al., 2003; Yagub et al., 2014; Ren et al., 2021; Olatunde et al., 2021; Omorogie et al., 2022;) has been used to treat dye-polluted wastewaters. Among all these, adsorption remains a favoured technique. This is due to its ease of use, cost-effectiveness, high removal efficiency, and environmental-friendliness. Adsorption employs plant and animal biomass, which are cheap and readily available, to remove toxic materials from wastewater. Different materials, including peanut hull (Brown et al., 2000),

corn cob (Fan et al., 2011), coconut leaf (Jawad et al., 2016), mungbean husk (Saeed et al., 2009), orange peel (Nascimento et al., 2014), maize seed chaff (Babalola et al., 2016d), algae (Elgarahy et al., 2019a), sepia shell (Elgarahy et al., 2019b), cellulose/clay composites (El-Aziz et al., 2018), are among the numerous biomass that have been previously used by other researchers for this purpose.

In addition, several of these pristine waste materials have been chemically modified by other researchers to improve their adsorption efficiency. Examples of such modifications are chemically modified orange waste (Lasheen et al. 2012), modified nanocomposites (Li et al., 2020), Fenton-activated biogenic waste (Bulut and Tez, 2007), Fenton-modified peanut hull (Lv et al., 2022), citrate-modified bagasse (Mpatani et al., 2020), acid-treated peanut shell (El-Shafey, 2007). These modification processes improve adsorption capacity of biomass through two major ways: (i) disrupting the lignin and cellulose structures in the biomass, giving room for more toxic chemicals to be sequestered, and (ii) introducing more active functional groups to the biomass, therefore, improving the interaction between the biomass and the toxic chemicals (Lv et al., 2022).

Herein, we present an efficient and eco-friendly modification method for improved sequestration of emerging contaminants from waste water. We employed the environmentally-friendly Fenton-like modification method, using recyclable nano FeOCl as Fenton chemical (Wang et al., 2020; Lv et al., 2022), to improve the sequestration potential of *Bauhinia tomentosa* seedpod for methylene blue (MB) removal from water. We studied the functional and morphological properties of the modified and unmodified adsorbents by Fourier transform infrared spectroscopy, Energy Dispersive X-Ray, and Scanning electron microscopy. In addition, we investigated the effect of different experimental conditions such as pH, dye concentration, adsorbent dose, and contact time, on their sorption capacity of the adsorbents, and analyzed their regeneration potential.

2. Materials and methods

2.1 Materials

Methylene blue dye was procured from Xilong Scientific Co., Ltd, China. Ferric chloride hexahydrate was purchased from Avondale Laboratory Ltd, England, while acetone, acetic acid, ethanol, and hydrogen peroxide were purchased from BDH chemicals Ltd, England. Sodium chloride, potassium hydroxide, hydrochloric acid and sodium hydroxide were acquired from Molychem, India. All reagents were of analytical grade and were used without further purifications.

2.2 Preparation of adsorbent

The *Bauhinia tomentosa* Linn seedpod (BTSP) used for this research was collected from Dominion University, Ibadan, Nigeria. Before collecting the samples, the flowering parts (seedpod, flowers, and leaves) of the plant was identified and authenticated at the herbarium unit of the Department of Botany, University of Ibadan, Nigeria (Authentication number UIH-23360). The seedpods were air-dried for four weeks until crisp and pulverized into fine powder using an industrial blender. The pulverized sample was sieved using a 100 μm sieve and either calcinated at 400 $^{\circ}\text{C}$ for 2 hrs and ground into fine powder to produce BTSP adsorbent, or Fenton-modified before calcination. Prepared sample was used immediately or stored at 4 $^{\circ}\text{C}$ in an air-tight container until use.

2.3 Fenton-like modification of adsorbent

Fenton-like modification of the pulverized and sieved seedpods was carried out according to already established protocol of Lv et al. (2022) with little modification. Briefly, 10 g of ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) was heated at 200 $^{\circ}\text{C}$ in an air-tight crucible for 2 hours in a muffle furnace. Unreacted ferric chloride was separated from the FeOCl nanosheets formed by repeated washing and centrifugation steps with acetone. 0.3 g of the FeOCl nanosheets and 5 g of the adsorbent material was added to a 250 mL beaker containing 100 ml of 2.5% H_2O_2 , and the mixture was heated at 50 $^{\circ}\text{C}$ for 6 hrs under constant agitation. The Fenton-like modified adsorbent was continuously washed with distilled water by centrifugation until a colourless and water clear filtrate was obtained. The modified adsorbent was dried at 70 $^{\circ}\text{C}$ for 2 hrs and calcinated at 400 $^{\circ}\text{C}$ for 2 hrs and ground into fine powder to produce the Fenton-BTSP adsorbent. Calcinated sample was used immediately or stored in the refrigerator at 4 $^{\circ}\text{C}$ until required.

2.4 Characterization of adsorbents

The functional groups present on the surface of BTSP and Fenton-BTSP was characterized by Fourier-transformed Infrared (FTIR) spectroscopy in the range of 4000 – 400 cm⁻¹ using a PerkinElmer FT-IR Spectrum 2 spectrophotometer (PerkinElmer, U.S.A.). The pH at which the overall charge on the surface of the adsorbents is zero, known as the pH-point of zero charge (pH-PZC), was investigated according to established protocol. The size of the adsorbents was measured by dynamic light scattering using a Zetasizer Nano S90 (Malvern instruments, U.K.). The surface morphology and elemental composition of the adsorbents was characterized by scanning electron microscopy (SEM) using a Hitachi SU 3500 scanning microscope (Hitachi, Japan) coupled to an energy dispersive X-ray (EDX) spectroscope at an accelerating voltage of 15 kV and resolution energy of 240 eV. Methylene blue absorbance was measured using a JENWAY 6305 UV-Vis spectrophotometer (JENWAY, U.K) at its wavelength of maximum absorption (662 nm). A more comprehensive protocol for each technique is provided as Supplementary Methods in the Supplementary Information.

2.5 Adsorption studies

The adsorption of MB onto BTSP or Fenton-BTSP was carried out using the batch adsorption technique according to established protocols (Babalola et al., 2017; Omorogie et al., 2022). All experiments were carried out in duplicates and the average was used in each case. For each experiment, 20 mL of 100 mg/L MB was measured into separate plastic bottles containing 20 mg of BTSP or Fenton-BTSP. The bottles were corked tightly and agitated at room temperature at 150 rpm and 2 hrs, while optimizing adsorption conditions such as dye concentration, pH, contact time, and adsorbent dosage. For pH studies, the pH of MB was adjusted to the range of 2.0–12.0 using 0.1 M NaOH or HCl. The effect of dye concentration was studied by varying MB concentration at a range of 25–500 mg/L. Adsorbent mass was varied between 10 mg to 500 mg to study the influence of adsorbent dosage on the sorption of MB. To study the effect of contact time on sorption efficiency of the adsorbents, aliquots were removed from the shaker at different time intervals of 0.5 min to 120 min. In all cases, supernatants were collected by centrifugation at 4000 rpm for 5 mins and analyzed for residual concentration of MB using a UV/Vis Spectrophotometer at 662 nm. The absorbances were converted to concentration using the slope and intercept obtained from a calibration curve of different MB concentrations, which is available in the supplementary information (Supplementary Fig. 1 (SF 1)). The quantity (q_e) of MB adsorbed by BTSP or Fenton-BTSP was calculated using Eq. 1.

$$q_e = C_o - C_e \times \frac{V}{W}$$

1

where q_e is the amount of MB adsorbed at equilibrium, C_o (mg/L) and C_e (mg/L) are the initial and final concentrations of MB, respectively, V (mL) is the volume of dye, and W (mg) is the weight of adsorbent.

2.6 Desorption studies

Desorption experiment was carried out using inorganic and organic acids and bases as desorbents. Briefly, 20 mg of adsorbent was shaken with 20 mL of 100 mg/L of MB solution at the pH of maximum adsorption for 2 hrs at room temperature. After separating the supernatant from the adsorbent by centrifugation, 20 mL of varying concentrations (0.01, 0.05, 0.1, 0.25, and 0.5 M) of HNO₃, KOH, acetic acid, or ethanol was added to the adsorbent and shaken for 30 mins at room temperature. The adsorbent was separated by centrifugation and the absorbance of the supernatant was analysed by UV-Vis at 662 nm. The percentage desorption (%D) was calculated using Eq. 2.

$$\%D = \frac{\text{amount desorbed}}{\text{amount adsorbed}} \times 100\% \quad (2)$$

2.7 Adsorption isotherm and kinetics

Four different isotherm models were used describe the adsorption equilibrium of varying concentrations of MB onto BTSP and Fenton-BTSP. Specifically, the adsorption data was fitted to the linear versions of Langmuir (Eqs. 3 and 4), Freundlich (Eq. 5),

Dubinin-Radushkevich (Eqs. 6, 7 and 8), and Temkin (Eq. 9) isotherm models. The linear pseudo-first order (Eq. 10), pseudo-second order (Eq. 11), and Elovich (Eq. 12) kinetic models were also employed to describe the mechanism of adsorption at different interaction times of MB with the adsorbent.

3

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m}$$

4

$$K_L = \frac{1}{1 + bC_o}$$

5

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$$

6

$$\ln q_e = \ln q_m - B_D \epsilon^2$$

7

$$\epsilon = \left[RT \ln \left(1 + \frac{1}{C_e} \right) \right]^2$$

8

$$E = \frac{1}{2B_D}$$

9

$$q_e = \gamma_T \ln K_T + \gamma_T \ln C_e$$

10

$$\ln(q_e - q_t) = \ln q_e + K_1 t$$

11

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e}$$

12

$$q_t = \alpha + \beta \ln t$$

where C_o (mg/L) and C_e (mg/L) are the initial and final concentrations of MB, respectively, q_t (mg/g) and q_e (mg/g) are the amounts of MB adsorbed at time t (min) and equilibrium, respectively, K_L (L/mg), K_F (mg/g), and K_T (L/g) indicate the affinity coefficient-related bonding terms, b (J/mol) is the Langmuir constant, n is a dimensionless exponent in the Freundlich equation, B_D (mol²/J) is related to the free energy of adsorption per mole of adsorbate, q_m (mg/g) is the maximum theoretical monolayer saturation capacity, E (J/mol) is the apparent energy, R is the universal gas constant (8.3142 J/mol/K), T (K) is the

absolute temperature, k_1 (min^{-1}) and k_2 (g/mg/min) are the first order and second order rate constants, respectively, α (mg/g/min) and β (g/mg) represent the constants related to the initial reaction rate and the activation energy, respectively.

3. Results and discussion

3.1 Characterization of adsorbents

The density of the bulk BTSP and Fenton-BTSP adsorbents are 0.68 and 0.72 g/cm^3 , respectively. The higher density of the Fenton-BTSP is probably due to the compact packing of iron that is present in the adsorbent. Elucidation of the adsorbents by FTIR revealed the presence of functional groups such as O-H, C = O, C-O, and C-N, as well as a Fe-O band at 568.23 cm^{-1} , which was more pronounced in the Fenton-BTSP (Fig. 1A). After adsorption of MB dye, some of these absorption frequencies exhibited substantial shifts or disappeared completely and new bands appeared. FTIR spectrum of the dye-loaded adsorbents are attached as Supplementary Figures in the Supplementary Information (SF 2 for BTSP and SF 3 for Fenton-BTSP), suggesting a chemical interaction between the adsorbents and MB molecules (Namasivayam et al., 1996; Babalola et al., 2017).

The pH-PZC of the surface of BTSP and Fenton-BTSP was found to be 5.548 and 7.655, respectively (Fig. 1B). The adsorbent is expected therefore, to acquire a positive surface charge below the pH-PZC in which the adsorption of anionic or acid dyes are enhanced, whereas above the pH-PZC, it acquires a negative surface charge thus favoring the adsorption of cationic or basic dyes. This indicates that the adsorption of MB, a cationic dye, should be optimum at a pH above the pH-PZC. Previous studies of MB have revealed that maximum adsorption was observed at a basic pH (Jawad et al., 2016; Mpatani et al., 2020).

Figure 1C shows the intensity-weighted size distribution of the adsorbent particles elucidated by DLS. The result showed different sizes of the adsorbents distributed mostly within the nanometer range. Both the BTSP and Fenton-BTSP showed similarity in their size distribution, which was averaged at ~ 60 nm and 200 nm, with larger, less-intense aggregated particles appearing within the micrometer range. This indicates that the adsorbents have very small (nano) sizes, which is an important criterion in selecting an adsorbent.

The scanning electron micrographs of the adsorbents at a magnification of 500x is shown in Fig. 2A-D. the micrograph shows agglomerated particles having irregular and rough surfaces with open pores and cavities. These cavities became loaded with MB dye (cloudy white patches) after adsorption (Fig. 2B & D). The adsorbent particles contain elements such as C, Si, O, Al, K, Na, Ca, Mg, and Fe (Table 1). The amount of Fe present was greatly increased ($\sim 10\times$) in the Fenton-BTSP adsorbent, confirming successful modification of the BTSP with the Fenton-like compound (FeOCl). Micrographs of the adsorbents in the presence or absence of MB obtained at higher magnifications are shown in the Supplementary Figures (SF 4 for BTSP and SF 5 for Fenton-BTSP).

Table 1
Elemental composition of BTSP and Fenton-BTSP adsorbents

BTSP				Fenton-BTSP			
Before adsorption		After adsorption		Before adsorption		After adsorption	
Element	Composition (%)	Element	Composition (%)	Element	Composition (%)	Element	Composition (%)
Carbon (C)	20.45	Carbon (C)	19.78	Carbon (C)	24.32	Carbon (C)	23.90
Oxygen (O)	34.09	Oxygen (O)	33.48	Oxygen (O)	31.85	Oxygen (O)	30.61
Aluminium (Al)	8.22	Aluminium (Al)	9.79	Silicon (Si)	17.62	Silicon (Si)	20.30
Silicon (Si)	29.17	Silicon (Si)	26.68	Aluminium (Al)	1.96	Aluminium (Al)	2.21
Calcium (Ca)	1.10	Calcium (Ca)	1.18	Nitrogen(N)	1.05	Nitrogen(N)	1.37
Sodium (Na)	3.34	Sodium (Na)	3.58	Potassium (K)	0.21	Potassium (K)	0.28
Magnesium (Mg)	1.98	Magnesium (Mg)	3.06	Iron (Fe)	22.89	Iron (Fe)	21.32
Iron (Fe)	1.65	Iron (Fe)	2.45				

3.2 Effect of different sorption conditions

Adsorption conditions such as pH, adsorbent dosage, dye concentration, temperature, contact time, among others, can greatly influence the rate and mechanism of adsorption. The effect of pH, adsorbent dosage, dye concentration, and contact time is shown in Fig. 3. By varying the pH of MB, maximum adsorption was obtained at a pH of 10 for both adsorbents with adsorption capacities of 60.94 mg/g and 72.80 mg/g for BTSP and Fenton-BTSP, respectively (Fig. 3A), indicating improved efficiency of the adsorbent after Fenton-like modification. This is consistent with previous reports (Lv et al., 2022). Adsorption was minimum at pH 2 with adsorption capacity of 2.41 mg/g for BTSP and at pH 4 with an adsorption capacity of 0.98 mg/g for Fenton-BTSP. The low rate of adsorption of MB by the adsorbents at acidic pH may be due to high proton density, which leads to ionic repulsion between the positively charged MB dye and the surface of the adsorbent (Omorogie et al., 2022). This is consistent with the deductions made from our pH-PZC experiment, and with previous adsorption experiments on MB (Jawad et al., 2016; Mpatani et al., 2020).

Variation of adsorbent dosage from 10–500 mg revealed a sharp decrease in adsorption capacity that is independent on Fenton modification. As the dose of the adsorbent increased from 10 mg to 500 mg, adsorption capacity witnessed a spontaneous decrease from 80.46 to 1.36 mg/g for BTSP and from 114.25 to 3.94 mg/g for Fenton-BTSP (Fig. 3B). Such sharp decrease in adsorption capacity at increased dosage can be associated with increased clogging or aggregation of adsorbent particles, which lead to increased diffusion path for MB, resulting in a substantially low rate of adsorption (Asgher and Bhatti, 2010; Babalola et al., 2016a).

The result of varying concentrations of MB on the adsorption efficiency of BTSP and Fenton-BTSP is shown in Fig. 3C. As the concentration of MB increases from 25 mg/L to 500 mg/L, the adsorption capacity of the adsorbents experienced a corresponding increase from 14.65 to 277.41 mg/g for BTSP and from 23.76 to 343.25 mg/g for Fenton-BTSP. This increase in adsorption capacity with increasing dye concentration is higher for the Fenton-BTSP, indicating improved efficiency due to modification with FeOCl, and can be associated with increased mass transfer driving force as the concentration of the dye increased. Such increase in mass transfer enhances the adsorption of more dye molecules by the adsorbent (Asgher and Bhatti, 2010; Babalola et al., 2016a; Omorogie et al., 2022).

To investigate the influence of adsorbent-dye contact time on the efficiency of adsorption, the time of adsorption was varied from 0.5 to 120 mins. The results (Fig. 3D) shows that the adsorption capacity of BTSP and Fenton-BTSP increased substantially with increasing contact time up to a level where equilibrium is reached. For both BTSP and Fenton-BTSP, adsorption was rapid for the first 30 mins after which further increase in contact time exerted no effect on the adsorption capacity of the adsorbents. This is probably because after 30 minutes, there are no cavities or unoccupied active surfaces available on the adsorbents for MB to occupy (Olatunde et al., 2021). Although Fenton modification of the adsorbent did not affect the time it took the adsorbent to reach equilibrium, the adsorption capacity was, however, greater for Fenton-BTSP than for BTSP.

3.3 Desorption of MB from adsorbents

Regeneration is an important factor in selecting adsorbents for wastewater management. In this study, the regeneration of BTSP and Fenton-BTSP was tested by agitating MB-loaded adsorbents with varying concentrations of organic and inorganic acids and bases for 30 mins. The desorption of MB from BTSP was maximum using an organic acid (acetic acid) at all operational concentrations, with 0.1 M acetic acid giving the highest yield of desorbed MB (21.18%) after 30 mins of agitation (Fig. 4A). In the case of Fenton-BTSP, the inorganic acid, HNO_3 desorbed the highest amount of MB at all operational concentrations, with optimal desorption (46.24%) observed using only 0.01 M of the acid (Fig. 4B). These findings are consistent with the results of our pH studies, and indicates that, counter ions from an acid are needed to remove the basic MB dyes adsorbed on the adsorbents. It is also worthy of mention that desorption of MB from the Fenton-BTSP was more efficient than from BTSP. As can be seen from Fig. 2A&B, the lowest amount of MB desorbed from BTSP was 3.53% using 0.05 M KOH. In comparison, the lowest amount of MB desorbed from the Fenton-BTSP was 17.38% using 0.1 M of KOH. Overall, desorption of MB was at least 2-folds higher in Fenton-BTSP compared to BTSP. This indicates that modification of the adsorbent with the Fenton FeOCl improved not only its adsorption property but also its regeneration and reusability potential.

3.4 Adsorption isotherm and kinetics

Equilibrium isotherm models such as the Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich models, were employed to explain the sorption process of MB onto BTSP and Fenton-BTSP. In all cases, the correlation coefficient (R^2) was used to choose the best fit model. In the case of isotherm modelling, the experimental data revealed that the adsorption of MB onto BTSP probably followed the Temkin isotherm model, which has the highest correlation factor ($R^2 = 0.6$) and an equilibrium binding constant (γ_T) of 41.85 L/mg (Table 2). This indicates that the heat of sorption of MB onto the surface of BTSP decreases linearly as the surface of the adsorbent is covered by the dye (Temkin and Pyzhev, 1940). The maximum monolayer adsorption capacity for BTSP revealed by Langmuir and Dubinin-Radushkevich models were 190.63 mg/g and 42.53 mg/g, respectively, with an apparent energy (E) of 2226.85 J/mol (Table 2). For Fenton-BTSP, adsorption of MB followed the Freundlich isotherm model with a correlation coefficient that approaches unity ($R^2 = 0.92$) with an equilibrium binding constant (γ_T) of 60.11 L/mg (Table 2). This suggests the formation of multiple layers of MB dye on the surface of the adsorbent improving its sorption capacity compared to the unmodified BTSP. The maximum monolayer adsorption capacity for Fenton-BTSP revealed by Langmuir and Dubinin-Radushkevich fittings were 520.83 mg/g and 158.71 mg/g, respectively, with an apparent energy (E) of 148.48 J/mol (Table 1). These results further establish that modification of BTSP with FeOCl nanosheets indeed improved its maximum adsorption capacity, which is consistent with previous findings (Lv et al., 2022). The isotherm model plots are shown in the Supplementary Figures (SF 6). When compared to other adsorbents used to sequester MB from aqueous solution (Table 3), BTSP and Fenton-BTSP showed outstanding capacities in removing MB from wastewater, with Fenton-BTSP displaying the greatest potential.

Adsorption kinetics describe the rate at which adsorbates are captured by the surface of the adsorbents (Unuabonah et al., 2017b). This information can be used to describe the dynamics and mechanism of dye uptake by the adsorbent. Here, linear kinetics models such as pseudo-first order, pseudo-second order, and Elovich models, were employed to describe the dynamics of MB uptake by BTSP and Fenton-BTSP (Table 4). Kinetics modelling of the adsorption data revealed that the

adsorption of MB onto BTSP and Fenton-BTSP could followed both pseudo-second order kinetics ($R^2 = 0.98$ for BTSP and 0.99 for Fenton-BTSP) and Elovich kinetics ($R^2 = 0.94$ for BTSP and 0.89 for Fenton-BTSP). However, the pseudo-second order model gave the best fit to experimental data as adjudged by the highest correlation factor (Table 4), indicating that the mechanism of adsorption of MB onto BTSP and Fenton-BTSP followed the chemisorption process, where the adsorption of MB by the adsorbents was controlled by electron transfer (Hosseini et al., 2021; Adeniji et al., 2019; Asgher and Bhatti, 2010; Olu-Owolabi et al., 2012). The extremely low correlation coefficient factor from the pseudo-first order model ($R^2 = 0.026$ for BTSP and 0.057 for Fenton-BTSP) suggests that the interaction between the MB dye and adsorbents is completely chemical and cannot be described by a physisorption mechanism. The kinetics model plots are shown in the Supplementary Figures (SF 7). The Elovich model revealed that the initial rate of adsorption of MB dye molecules onto the surface of the adsorbents experienced over 2-fold increase on modification with FeOCl. As can be seen in Table 3, the initial reaction rate (α) is 4.34 mg/g/min for BTSP and 11.98 mg/g/min for Fenton-BTSP. This further suggests that Fenton modification of adsorbents can be a potent way of improving their capacity for dye sequestration and wastewater management.

Table 2
Linear isotherm parameters for the adsorption of MB onto BTSP and Fenton-BTSP

Model	Parameters	BTSP	Fenton-BTSP
Langmuir	q_m	190.63	520.83
	R_L	0.87	0.49
	b	0.0011	0.010
	R^2	0.17	0.70
Freundlich	K_F	1.11	15.78
	$1/n$	0.82	0.56
	R^2	0.58	0.92
Temkin	γ_T	41.85	60.11
	K_T	0.34	0.36
	R^2	0.60	0.64
Dubinin-Radushkevich	q_m	42.53	158.71
	B_D	1.01E-07	2.27E-05
	E	2226.85	148.48
	R^2	0.011	0.74

Table 3
Comparison of adsorption capacities of some adsorbents on MB.

Adsorbents	Modification	q_m (mg/g)	References
Rice straw	-	158.0	Jawad et al., 2020
Coconut leaf	-	112.3	Jawad et al., 2016
Peanut hull	-	64.0	Lv et al., 2022
Passion fruit waste	-	44.7	Pavan et al., 2008
Dragon fruit peels	-	192.3	Jawad et al., 2018
<i>Bauhinia tomentosa</i> seedpod (BTSP)	-	190.63	This work
Fenton-like modified Peanut hull	FeOCl	132.4	Lv et al., 2022
Citrate-modified sugarcane bagasse	Citrate	224.0	Mpatani et al., 2020
Silica-modified Soya waste	Silica	47.0	Batool and Valiyaveetil, 2021
Base-modified pine corn	NaOH	33.7	Yagub et al., 2014
Acid-modified defatted algae	H ₂ SO ₄	7.8	Chandra et al., 2016
Fenton-like modified <i>Bauhinia tomentosa</i> (Fenton-BTSP)	FeOCl	520.83	This work

Table 4
Linear kinetics parameters for the adsorption of MB onto BTSP and Fenton-BTSP

Model	Parameters	BTSP	Fenton-BTSP
Pseudo-first order	K_1	0.040	0.047
	q_e	2.86	7.73
	R^2	0.026	0.057
Pseudo-second order	K_2	0.0046	0.0028
	q_e	26.39	71.74
	R^2	0.98	0.99
Elovich	α	4.34	11.98
	β	4.72	16.041
	R^2	0.94	0.89

4. Conclusion

We report an easy modification method for adsorbents for improved uptake of emerging pollutants from wastewater. This modification method, involving the Fenton-like chemical, FeOCl, improved the surface morphology, functional composition, and overall efficiency of a cost-effective and easy-to-get plant waste from *Bauhinia tomentosa* to rapidly remove methylene blue (MB) dye from water with a maximum adsorption capacity of 520.83 mg/g as compared to 190.63 mg/g obtained for the unmodified adsorbent. We discovered that although the adsorption equilibrium time was not affected by modification, the rate of the adsorption of MB was increased by over 2-folds due to the Fenton-like modification. The unmodified (BTSP) and Fenton-like modified (Fenton-BTSP) adsorbents responded at different extents to differences in solution pH, dye concentration,

adsorbent dosage, and reaction time. In addition to an improvement in the adsorption capacity on modification, it also appears that the regeneration of the adsorbents was greatly improved after modification. Modification of BTSP with FeOCl improved its surface morphology and functional composition in such a way that allows uptake of multilayers of MB dye on its surface via a chemisorption process, which explains the basis for its improved efficiency. It is therefore recommended that adsorbents be modified with the Fenton-like agent, FeOCl, for improved capacity and reusability in wastewater management.

Declarations

Author's contribution

M. T. Agbadaola conceptualized the idea in this manuscript, supervised this work as part of D.A. Olatunji's BSc research project, generated some of the results contained in this work at the Institute of Molecular Biosciences, Karl Franzens Universität, Graz, Austria, analyzed all the data, wrote the first manuscript, revised, and reviewed the manuscript. *D. A. Akinyemi* co-supervised this work, revised and reviewed the manuscript. *D. A. Olatunji* generated some of the data in this manuscript as part of her BSc research project. *J. O. Babalola* conceptualized the idea in this manuscript, revised and reviewed the manuscript. All authors approved the final manuscript for publication.

- **Competing Interests**

The authors declare that there are no conflicts of interest.

- **Funding**

No funding was received for conducting this study.

Acknowledgement

M. T. Agbadaola acknowledges Prof. Sandro Keller at the Institute of Molecular Biosciences, Karl Franzens Universität, Graz, Austria, for allowing him to use his laboratory facility to generate some of the results that make up this manuscript.

Data availability

All data used to produce this manuscript will be made available upon request to the corresponding author.

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Figures

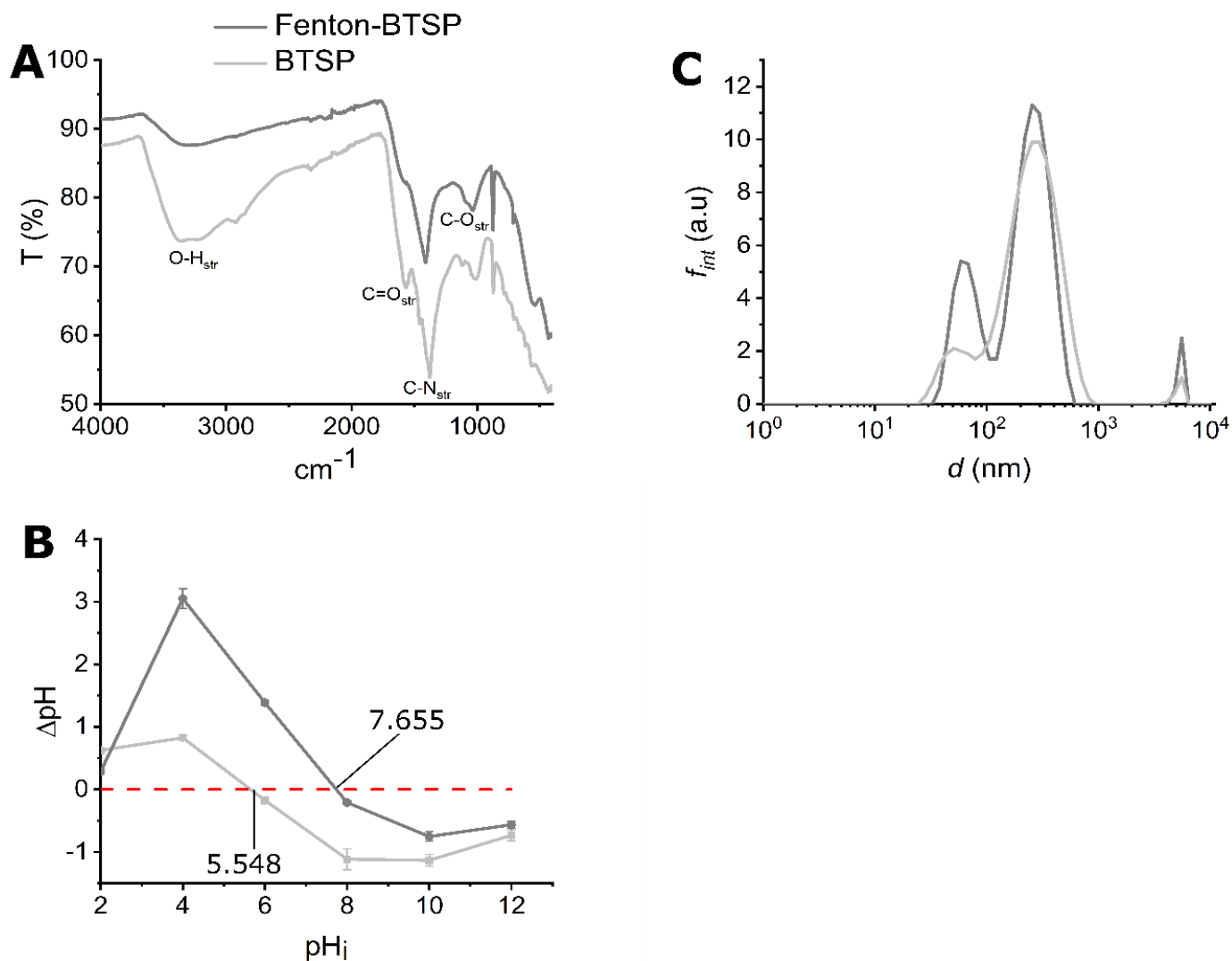


Figure 1

Characterization of adsorbents. (a) FTIR spectra of BTSP and Fenton-like modified BTSP. (b) pH-PZC plot of BTSP and Fenton-BTSP. (c) Size distribution of BTSP and Fenton-BTSP elucidated by DLS. All measurements were carried out at 298 K. Error bars are standard errors of two independent experiments.

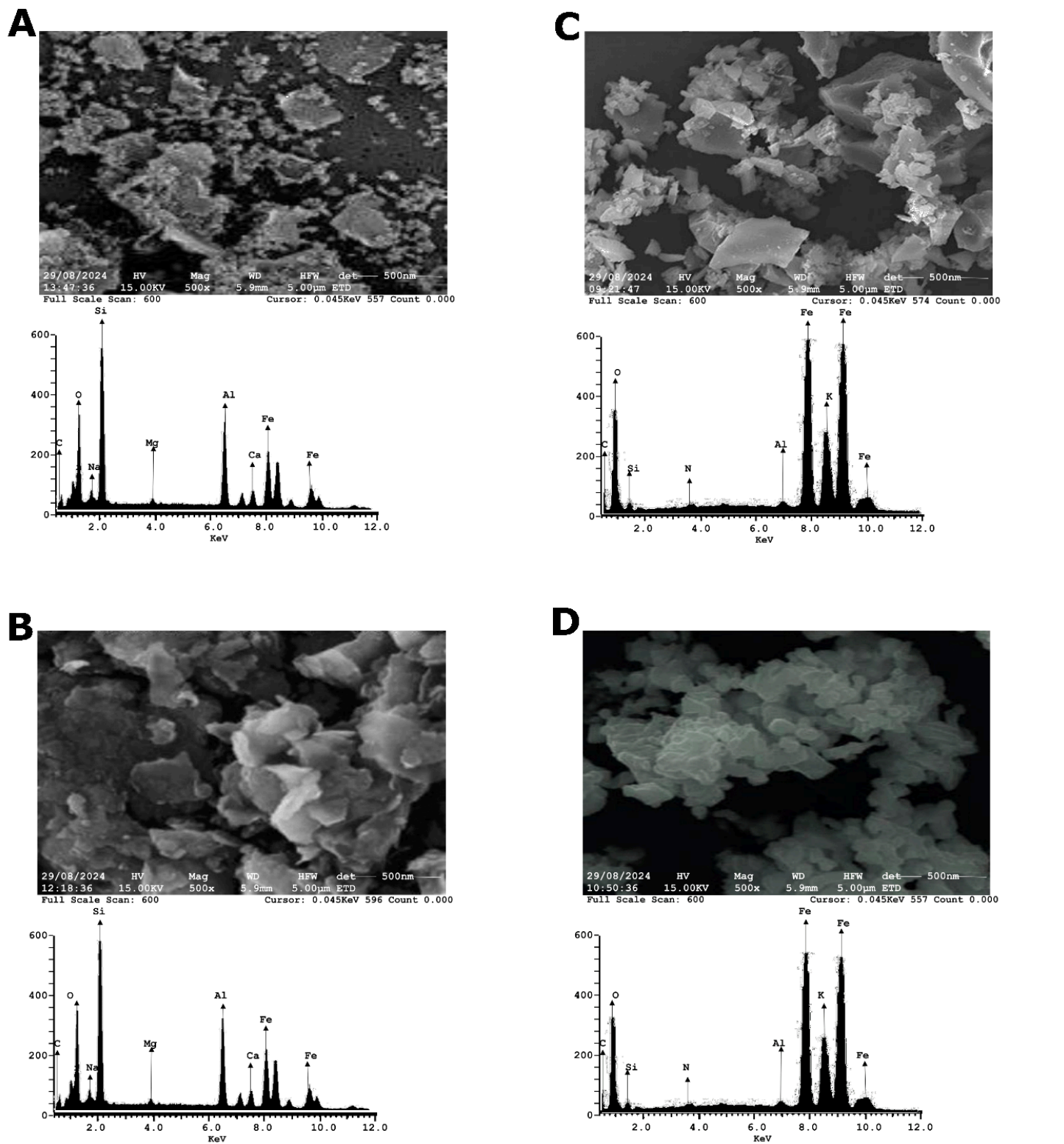


Figure 2

Surface morphology of adsorbents. (a) SEM-EDX spectra of BTSP at 500x before adsorption. (b) SEM-EDX spectra of BTSP at 500x after adsorption of MB. (c) SEM-EDX spectra of Fenton-BTSP at 500x before adsorption. (d) SEM-EDX spectra of Fenton-BTSP at 500x after adsorption of MB.

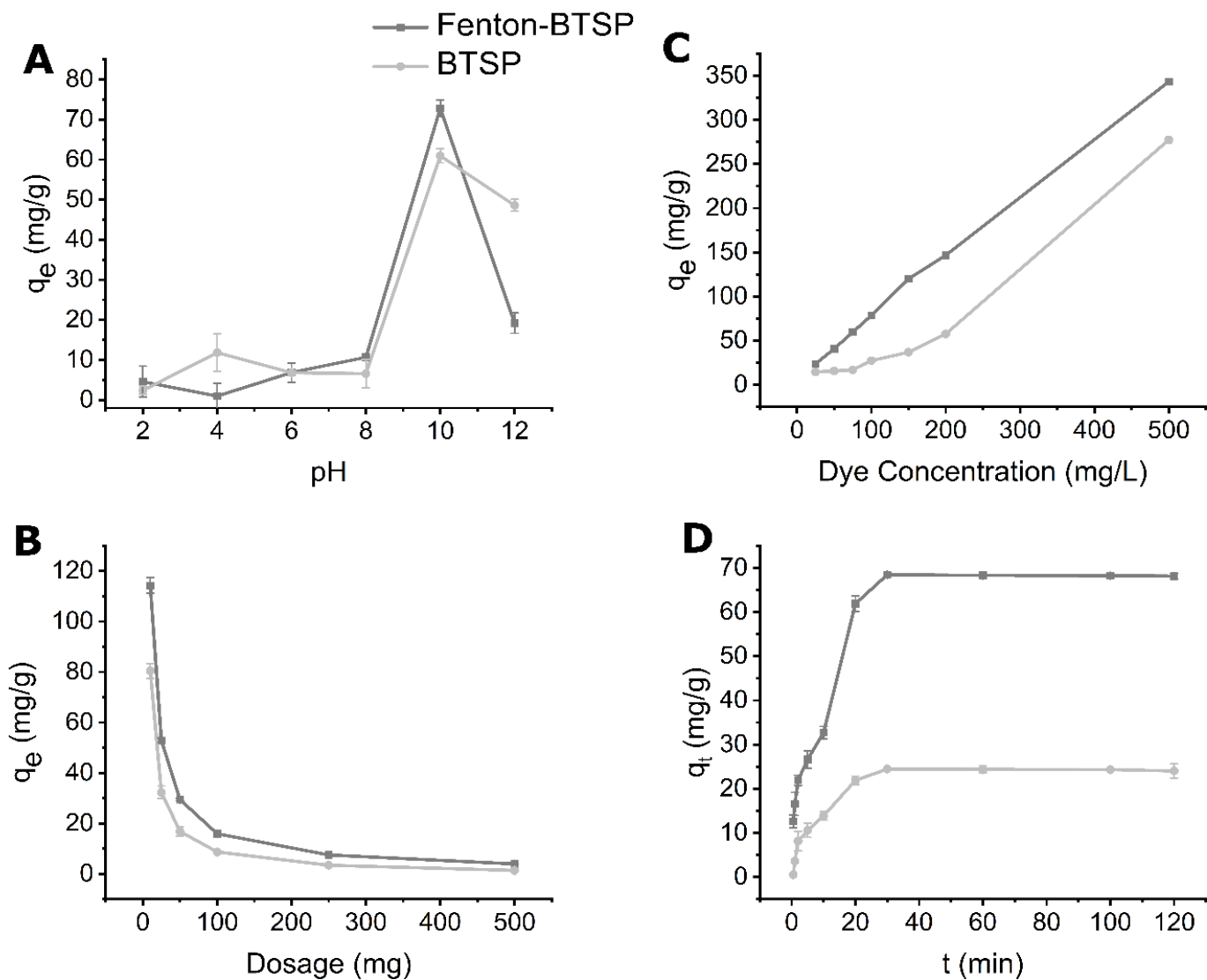


Figure 3

Influence of different sorption conditions on the adsorption of MB onto BTSP and Fenton-BTSP. (a) Effect of pH (b) Effect of adsorbent dosage. (c) Effect of MB concentration. (d) Effect of contact time. Error bars are standard errors of two independent experiments.

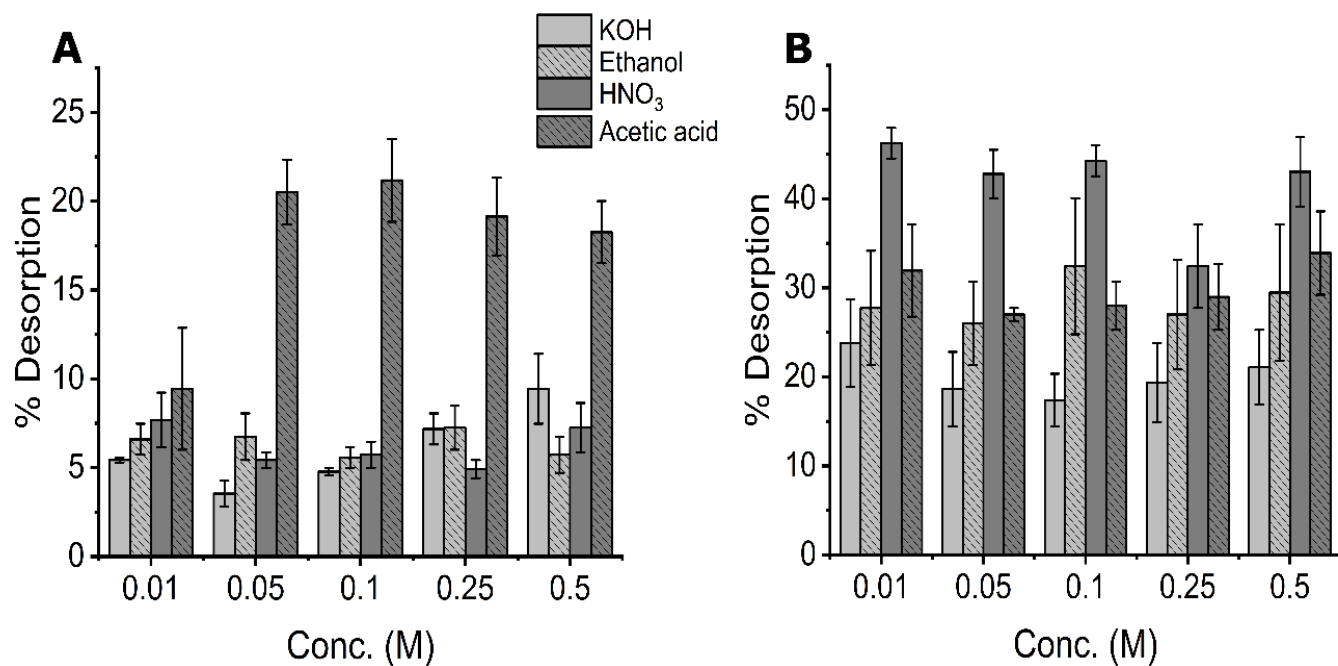


Figure 4

Desorption of MB from BTSP and Fenton-BTSP using organic and inorganic acids and bases. (a) Desorption of MB from BTSP. (b) Desorption of MB from Fenton-BTSP. Adsorbent dose = 20 mg; initial dye concentration = 100 mg/L; temperature = 298 K; desorption time = 30 min; agitation speed = 150 rpm. Error bars are standard errors of two independent experiments.

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