Color removal from anaerobic/aerobic treatment effluent of bakery yeast wastewater by polyaniline/beidellite composite materials

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ABSTRACT

The adsorption technique is widely applied for the removal of pollutants from wastewater, especially for toxic or non-biodegradable wastewater. In recent years, the production of alternative adsorbents to replace costly adsorbents has been paid more attention to in literature. Polyaniline/beidellite (PAn + Bei) composite material as an absorbent, which is efficient and low cost can easily be prepared via H2SO4, KIO3, and aniline. This paper deals with color and total organic carbon (TOC) removal of biologically treated bakery yeast wastewater (BYW) using the PAn + Bei composite material by adsorption processes. The effects of experimental variables were chosen as the initial pH (pHs), sorbent dosage (m), contact time (t), and mixing speed (s) by a batch sorption process. It was found that by increasing the adsorbent dosage (0.025–0.400 g/50 ml of composite dosage), contact time (2–240 min) and decreasing the pH (9–3) improved the color and TOC removal efficiencies. The optimum color and TOC removal efficiencies were obtained as 88.7% and 63.3% at 0.400 g/50 ml of adsorbent dosage, a pH of 3, 240 rpm, and 240 min. In addition, a pseudo-second-order kinetic model was proposed to correlate the experimental data. To understand the removal mechanism and characterize the surface of the PAn + Bei composite material, size exclusion chromatography (SEC), BET surface analysis, fourier transform infrared spectroscopy (FTIR) analysis, scanning electron microscope (SEM), thermal gravimetric analysis (TGA), and differential scanning calorimetry (DSC) were employed. As a consequence, the proposed mechanism for the removal by PAn+Bei composite material seems to be driven by an ion exchange process.

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1. Introduction

Baker’s yeast production by fermentation uses molasses as a raw material which is a by-product of the sugar industry. Due to the molasses, baker’s yeast wastewater (BYW) has a high organic content and dark brown color. Melanoidsins are the source of this dark brown color in BYW which is resistant to biodegradation. Melanoidsins restrict the sunlight and make a reduction in the natural photochemical process for self-purification of the surface waters [1,2].

Various treatment methods such as a biological process (anaerobic, aerobic), physico-chemical treatment (adsorption, membrane process, reverse osmosis, coagulation/floculation, electrocoagulation) and oxidation processes (ozone, Fenton) have been performed for the treatment of BYW [3,4]. Nowadays biological treatment of BYW is realized by combinations of anaerobic digestion and aerobic systems that successfully reduce BOD and COD to acceptable limits, but do not deal effectively with the dark color and limits the reuse/recycling of the process water.

Treatment by oxidation technology is generally effective on the color, but not COD, membrane filtration processes are prone to fouling [5,6], and reverse osmosis generates a high salinity that presents disposal difficulties [7]. Coagulation removes color and COD effectively, but possesses a number of drawbacks such as necessities for high quantity of inorganic coagulants. Decolorization through chemical treatment by ozone, Fenton’s reagent and H2O2/UV lead to temporary color reduction because of the transformation of the chromophores [8–11].

For these reasons, the BYW requires an alternative treatment technology which is relatively simple and effective in the removal of color before its safe disposal into the environment. Adsorption techniques are widely used to remove pollutants from the water, especially those that are toxic and not easily biodegradable. The studies focused on the production of alternative adsorbents to replace the costly ones reported in literature. Attention has focused on composite materials which are generally produced with various natural solids such as beidellite, cheap natural clay, and polymeric materials like polyaniline (PAn) for the effective removal of
pollutants when compared with other polymeric materials. PAN has a large variety of advantages such as high removal efficiencies, low cost, easy synthesis, both chemically (powder form) and electrochemically (a film), and environmental stability. Thus, studies have focused on the production of PAN coated different composite materials recently [12,13]. In literature, sawdust [14–17], rice husk and saw dust of Eucalyptus camaldulensis [18], palygorskite [19], montmorillonite [20], silica gel [21] were coated by polyaniline for the removal of pollutants from water/wastewater.

The novelty of this study is the production of a new composite material by coating beidellite with aniline to use it for color and TOC removal of biological treated baker’s yeast wastewater for the first time. The effects of the variables such as initial pH (pHs), sorbent dosage (m), contact time (t) and mixing speed (s) on the adsorption were examined by using a batch method. Several kinetic mechanisms were applied on data and molecular weight distributions (MWDs) were monitored during treatment by SEC. SEM, TGA, BET surface analysis, DSC, and FTIR were used to characterize the PAN + Bei composite materials.

2. Materials and methods

2.1. Preparation of beidellite/polyaniline composite

Beidellite is a dioctahedral smectite named after Beidell, CO, USA. Large deposits of beidellite minerals were explored in different locations of Turkey [22]. It was used for daily cleaning processes in the Black Sea region of Turkey. PAN is a poly aromatic amine that can be easily synthesized chemically from Brønsted acidic aqueous solutions. It is one of the most potentially conducting polymers and has obtained considerable attention in recent years. Chemical polymerization of aniline in aqueous acidic media (Brønsted acid) can easily be performed by the use of chemical oxidizing agents such as (NH4)2S2O8, KIO3 and K2Cr2O7 as shown in Fig. 1 [23]. There are a few studies regarding the production of polyaniline composites material in which different coated material, oxidizer, acid types and production processes were used. Ansari [14] and Ghorbani et al. [24], have produced different polyaniline composite materials by similar production processes with this study. Ghorbani et al. [24], produced the polyaniline and polypyrrole composites material for treatment of a real wastewater, paper mill wastewater, and the results showed that polyaniline/polypyrrole composite material have an important potential for using pollutants from real wastewater. In this study, preliminary studies were realized according to literature, then the production conditions of Ansari [14] and Ghorbani et al., were modified for production of PAN + Bei composite materials. For preparation of composite materials, 1 g of beidellite was added to 100 ml H2SO4 (1 M) solution and the solution was mixed at a stirring rate of 500 rpm [25]. Then, 1 g KIO3 was added at room temperature and 1 ml of aniline monomer was injected slowly. The polymerization was carried out at room temperature for 2 h. In order to remove the unreacted monomers and oxidants, the final product was washed thoroughly with 500 ml of deionized water. Then, the product was dried at 60 °C for 24 h. The beidellite was used as received without any pretreatment.

2.2. Experimental set-up and analytical methods

The anaerobic-aerobic treated wastewater was obtained from the baker yeast production industry in Turkey and batch adsorption tests were carried out in a NUVE ST–402 model shaker. TOC levels were determined through combustion of the samples at 680 °C using a non-dispersive IR source (Shimadzu, TOC-L model). The pH of the sample was adjusted with H2SO4 or NaOH and measured by a pH meter (WTW Inolab pH 720) and the color of the wastewater was measured utilizing a UV–Vis spectrophotometer at 475 nm (PerkinElmer 550 SE).

JEOL 6.060 type scanning electron microscopy (SEM) was used to obtain SEM images. The FTIR spectra of the beidellite and PAN + Bei composite material were recorded on a PerkinElmer Spectrum 100. The TGA curves were performed on a Mettler Toledo TGA 1 Star System at a heating rate of 15 °C/min from room temperature to 1000 °C. For DSC analysis, a Mettler Toledo DSC1 instrument was used. The BET surface area was measured from the N2 adsorption/desorption isotherms with a Micromeritics ASAP 2010 analyzer (US).

The MW was determined using HPSEC with a Hewlett-Packard HPLC 1100-series system equipped with a refractive index (RI) detector, a diode array detector (DAD) and two ultrahydrogel (Waters, Product Number: WAT011535 and WAT011525) columns. RID measures change in refractive index and its unit was called “Refractive Index Units (nRIU)”. DAD is a type of UV–vis detector and its unit was named as absorbance unit (AU) at any wavelength. Deionized water at a flow rate of 1 ml/min was used as the mobile phase. The results were calibrated using 1 g/l of polyethylene oxide with different molecular weights (25.3, 44.0, 78.3, 152.0 and 326.0 kDa). The weight-average molecular weight (Mw) and number-average molecular weight (Mn) were calculated from the data using the following equations [26] and these calculations were processed by ChemStation software.

\[
M_w = \frac{\sum n_i M_i^2}{\sum n_i M_i}
\]

\[
M_n = \frac{\sum n_i M_i}{\sum n_i}
\]

Mw and Mn are the molecular weight and the height of each ith fraction eluted at the ith volume in the chromatogram, respectively. The polydispersity index (PDI) or heterogeneity index, is a measure of the distribution of molecular mass in a given polymer sample. PDI is calculated from \(M_w/M_n\).

Fig. 1. Overall polymerization reaction of polyaniline [23].
2.3. Adsorption kinetics

The percentage removal of TOC and the adsorption capacity of adsorbents (mg/g) were calculated using the following relationships:

\[ \text{TOC removal(\%) } = \frac{100(C_i - C_f)}{C_i} \]  

(3)

The adsorption capacity of adsorbents, \( q_e \), is calculated as:

\[ q_e = \frac{(C_i - C_f)V}{m} \]  

(4)

where \( m \) is the mass of PAN+Bei (g), \( V \) (l) is the volume of wastewater. \( C_i \) and \( C_f \) are the initial and final concentrations (in mg/l) of TOC, respectively.

The study of adsorption kinetics describes the solute uptake rate and evidently this rate controls the residence time of adsorbate uptake at the solid–solution interface. The adsorption rate constants for the TOC removal were calculated by using pseudo-first order, second-order and intraparticle diffusion kinetic models [27] which were used to describe the mechanism of the adsorption. The accord between the experimental data and the model-predicted values were expressed by the correlation coefficients (\( R^2 \)). A relatively high \( R^2 \) value indicates that the model successfully describes the kinetics of the adsorption.

2.3.1. The pseudo-first-order equation

A pseudo-first-order equation can be expressed in a linear form as:

\[ \log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303}t \]  

(5)

where \( q_e \) and \( q_t \) are the amount of TOC adsorbed (mg/g) on the adsorbents at the equilibrium and at time \( t \), respectively, and \( k_1 \) is the rate constant of adsorption (min\(^{-1}\)). Values of \( k_1 \) were calculated from the plots of \( \log(q_e - q_t) \) versus \( t \).

2.3.2. The pseudo-second-order equation

The pseudo-second-order adsorption kinetic rate equation is expressed as:

\[ \frac{dq_t}{dt} = k_2(q_e - q_t)^2 \]  

(6)

where \( k_2 \) is the rate constant of pseudo-second-order adsorption (g/mg min\(^{-1}\)). By integrating and applying the initial conditions, we have a linear form as:

\[ \frac{t}{q_t} = \frac{1}{k_2q_e^2} + \frac{1}{q_e}t \]  

(7)

where \( q_e \) is the amount of TOC adsorbed at equilibrium (mg/g). The second-order rate constants were used to calculate the initial sorption rate, \( h = k_2q_e^2 \). Values of \( k_2 \) and \( q_e \) were calculated using the intercept and the slope of the linear plots of \( t/q_t \) versus \( t \).

2.3.3. Intraparticle diffusion

The rate constant for intraparticle diffusion (\( k_{id} \)) is calculated using following equation:

\[ q = k_{id}t^{1/2} + C_i \]  

(8)

where \( q \) is the amount TOC adsorbed (mg/g) at time \( t \), \( k_{id} \) (mg/g min\(^{1/2}\)) is the rate constant for intraparticle diffusion and \( C_i \) is the thickness of the boundary layer. The values of \( k_{id} \) were calculated from the slope of the linear plots of \( q \) versus \( t^{1/2} \) [28,29].

3. Results and discussion

3.1. Morphology and characterization

In this study, a scanning electron microscope (SEM) was used to characterize the surface of the PAN+Bei composite material at a very high magnification and an accelerating voltage of 15 kV. Samples were coated with gold using a sputter coater with conductive materials to improve the quality of the micrograph. The morphology of beidellite and PAN+Bei composite material was illustrated in Fig. 2. The coating by conducting polymer by surface polymerization was very visible.

The BET specific surface area, Langmuir surface area and adsorption average pore width were calculated as 66.53, 89.48 m\(^2\)/g, and 4.95 nm for beidellite and 16.12, 22.74 m\(^2\)/g, and 17.94 nm for PAN+Bei composite material. During polymerization, some of the pores in the beidellite were stocked by PAN particles, so PAN+Bei had a smaller surface area than beidellite. Small surface areas and higher removal efficiencies by PAN+Bei showed that the removal mechanism by PAN+Bei depended on the ions exchange process. In literature, the main removal mechanism by polyaniline composite materials was reported as the ions exchange process [14,30–33].

The chemical structure of the composite material was determined by FTIR spectrum (Fig. 3) which was a significant tool to distinguish the characteristics of the functional groups. Small amounts of silica species such as amorphous material or quartz were indicated by the presence of a minor band around 836 cm\(^{-1}\). The octahedral Al—OH groups in the beidellite structures were reported around 911 cm\(^{-1}\). The infrared spectra of the beidellite were very similar in the Si—O region around 1000 cm\(^{-1}\). The interlayer water in the beidellite samples was characterized by an \( \text{H}_2\text{O} \) bending vibration at 1635 cm\(^{-1}\). The shoulder around 3240 cm\(^{-1}\) was ascribed to interlayer \( \text{H}_2\text{O} \) chemically bound or coordinated to the interlayer cation. The band at about 3618 cm\(^{-1}\)
The characteristic absorption peak of Al—O—Si for beidellite was observed at 983 cm⁻¹, which merged into a strong peak at 1127 cm⁻¹ of PAn+Bei [35]. The peaks at 792, and 1243 cm⁻¹ were reported as C—H out of plane, and C=N stretching vibrations, respectively [18]. The peaks at 1558 and 1486 cm⁻¹ for PAn+Bei were usually representative of the C=C stretching vibrations of quinone and benzene rings [24,35]. On the other hand, the peak at 1296 cm⁻¹ was correspondent to the C—N stretching vibration [24]. The FTIR analysis confirmed that PAn was successfully grafted on the surface of beidellite.

TGA curves for beidellite, and PAn + Bei were presented in Fig. 4. There were three main mass loss steps observed for beidellite. The first mass loss (5.5 wt%) occurring from room temperature to 100 °C was related to the loss of adsorbed water. Between 100 and 200 °C, a maximum weight loss (9.5 wt%) was realized, attributed to the removal of interlayer water and the onset of dehydroxylation. The dehydroxylation reached a low level of loss (4.0 wt%) between 400 and 750 °C. Similarly, TGA curves for PAn + Bei showed the weight loss of 4.9% up to 100 °C due to the presence of moisture. The weight loss (50.1 wt%) between 100 and 1000 °C mainly corresponded to the removal of interlayer water, the dehydroxylation and the decomposition of polymer coverage on the beidellite. The presence of excess water in beidellite and PAn + Bei was also confirmed by the FTIR spectrum.

The differential scanning calorimetry (DSC) curve (Fig. 5) showed that an endothermic reaction took place between 50 and 250 °C corresponding to water loss from the beidellite surface and interlayered space. On the other hand, there were three different conditions observed for PAn + Bei; an endothermic reaction between 50 and 140 °C, an exothermic between 140 and 314 °C, and an endothermic reaction between 314 and 400 °C. The endothermic reaction between 50 and 140 °C corresponded to water loss from the surface and the interlayered space of composite material was observed. The presence of PAn caused an exothermic reaction between 140 and 314 °C and above 314 °C a second endothermic reaction was present corresponding to the beginning of the collapse of the interlayered structure [36].

3.2. Effect of variables on removal efficiencies

In the adsorption process, pH, pollutant concentration, pollutant types, temperature, adsorbent dosage, operating time, mixing speed, particle size are some of the effective process parameters. In this study, the effects of pH, adsorbent dosage, mixing speed, and operating time were investigated on color and TOC removal efficiencies as discussed below.

pH is one of the important parameters that affect the interaction between the adsorbent and adsorbate [37]. In addition, pH affected the polymerization degree of melanodins
Under with the neutral decrease adsorption nitrogen removal [15,16]. The examined results were realized in acidic conditions, where the adsorption took place [41]. It was clear that increasing the adsorbent dosage (0.025–0.400 g/50 ml of composite dosage) improved the removal efficiencies from 62.5% to 88.8% for color and from 9% to 42% for TOC as seen in Fig. 7.

Due to the solute distribution in the bulk solution and external boundary film formation, it was reported that the mixing speed had an impact on adsorption efficiencies [42]. In this study, the effect of mixing speed on adsorption efficiencies was examined in the range of 40–240 rpm during an operating time of 120 min. As seen in Fig. 8, the mixing speed of 240 rpm was appropriate for the performed study.

The results for effect of contact time were shown in Figs. 6–8. As shown in Figs. 6–8, PAN+Bei composite material rapidly removed color and TOC in less than 30 min due to the abundant availability of active binding sites on the sorbent, and with gradual occupancy which was the source of color in BYW. Decreasing pH caused a decrease of color intensity [38]. Thus, the treatment of BYW was examined at different pH, and the results were presented in Fig. 6. The results showed that the removal of BYW by PAN+Bei composite material was higher in acidic conditions than those in neutral and alkaline. In literature, two possible pollutants removal mechanism were reported via polyaniline composite materials. If wastewater contains positive ions, the effective removal was realized under neutral and alkaline conditions [15,16]. On the other hand, if wastewater contains negative ions, the effective removal was realized under acidic conditions [18]. Under acidic conditions the nitrogen atom present in the PAN+Bei composite material was protonated. As a result, the higher adsorption behavior of the composite was explained by the electrostatic interaction between this protonated polymer and pollutants. However, as the pH increased (5–9) deprotonation of nitrogen atoms occurred leading to the depletion of active sites in the polymer skeleton. Therefore, the interaction of composite with the pollutant molecule was hindered and consequently results were in the lower adsorption. This result is in agreement with literature [18,37,39].

As seen in Fig. 3, when pH was increased from 3 to 9, the removal efficiencies decreased from 82.6% to 24% for color and from 21.8% to 6.5% for TOC. The arrangement of wastewater for <pH 3 by H₂SO₄ was a major cost item in the calculation of the operating cost as reported in the previous study [40]. Due to its high cost, the experimental conditions (<pH 3) were not considered.

Commonly, the increase of adsorbent dosages improved adsorption efficiencies. This could be explained by the increase in adsorbent dosages and surface area, where the adsorption took place [41]. It was clear that increasing the adsorbent dosage (0.025–0.400 g/50 ml of composite dosage) improved the removal efficiencies from 62.5% to 88.8% for color and from 9% to 42% for TOC as seen in Fig. 7.

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3.3. Kinetic studies and intra-particle diffusion models

In this study, pseudo-first order, pseudo-second order and the intra-particle diffusion model were tested with the results to obtain kinetics of the adsorption processes and mass transfer models. The constants and correlation coefficient ($R^2$) values for pseudo-first order, pseudo-second order and intra-particle diffusion model with respect to pH, $m_0$ (g) and $S$ (rpm) were given in Table 1. The linear correlation values close to 1 showed that the model was statistically significant and indicated the applicability of these kinetic equations.

All $R^2$ values for pseudo-second order were >96%. However, $R^2$ values for the pseudo-first order and the intra-particle diffusion model were varying at a high range. The data fitting for TOC illustrated that the pseudo-second order model fitted well and suggested chemical sorption as the rate-limiting step of the adsorption mechanism and no involvement of a mass transfer in solution [43].

In literature, the studies which dealt with the preparation of polyaniline composite materials generally focused on the removal of synthetic wastewater. On the other hand, Ghorbani et al. [24], produced the polyaniline composites material for treatment of real wastewater, paper mill wastewater, and reported that the complete removal of COD was not possible by adsorption with polyaniline composite materials. However, Ghorbani and Eisazadeh [44], reported >97% of COD removal for cotton textile wastewater by using polyaniline nanocomposites coated on rice husk ash. These results showed that the composite types and wastewater content affect the removal efficiencies. The best color and TOC removal efficiencies for anaerobic/aerobic treated baker yeast wastewater were reported as 86% and 43% by electro-coagulation process in our previous study [2], which were the similar to the results of this study. This similarity showed that PAN + Bei composite material had an important potential in the treatment of anaerobic/aerobic treated baker yeast wastewater.

3.4. The molecular weight distributions of treated BYW

The HPSEC method was able to successfully used to detect the MWDs of BYW. In HPSEC, larger molecules move rapidly through the column with eluent and leave the column faster than the smaller ones. The molecular weight distributions of colorful components (Fig. 9) were obtained by the DAD detector and it was clear that there was a remarkable removal of colorful components in wastewater.

<table>
<thead>
<tr>
<th>Table 1</th>
<th>The model parameters of pseudo first order, pseudo second order and intra-particle diffusion.</th>
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<tbody>
<tr>
<td>Pseudo first order</td>
<td>Pseudo second order</td>
</tr>
<tr>
<td>$q_{e,cal}$</td>
<td>$k_1$</td>
</tr>
<tr>
<td>pH</td>
<td>3</td>
</tr>
<tr>
<td>4</td>
<td>3.1</td>
</tr>
<tr>
<td>5</td>
<td>2.9</td>
</tr>
<tr>
<td>6</td>
<td>1.9</td>
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<tr>
<td>7</td>
<td>-</td>
</tr>
<tr>
<td>9</td>
<td>1.6</td>
</tr>
<tr>
<td>$m_0$ (g)</td>
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</tr>
<tr>
<td>0.05</td>
<td>7.3</td>
</tr>
<tr>
<td>0.1</td>
<td>4.1</td>
</tr>
<tr>
<td>0.3</td>
<td>2.4</td>
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<tr>
<td>0.4</td>
<td>1.8</td>
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<tr>
<td>$S$ (rpm/min)</td>
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<tr>
<td>120</td>
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</tr>
<tr>
<td>160</td>
<td>5.3</td>
</tr>
<tr>
<td>240</td>
<td>7.2</td>
</tr>
</tbody>
</table>

* Satisfactory positive log($q_e$-$q_i$) values was not available at pH 7.
In addition, $M_p$ values increased gradually by contrast with Mn, and BWV has the smallest PDI value for raw wastewater (Table 2). These results showed that PAn + Bei composite material preferentially removed the lower molecular weight components.

### 4. Conclusions

In this study, beidellite was coated with polyaniline to obtain low cost and effective adsorbent. The effects of pH, contact time, adsorbent dosage, and mixing speed were studied for the removal performance of the composite material on color and TOC removal efficiencies. The results showed that polyaniline coated beidellite had an important potential for use in wastewater treatment. The optimum color and TOC removal efficiencies were obtained as 88.7% and 63.3% at 0.400 g/50 ml of absorbent dosage, pH of 3, 240 rpm, and 240 min. SEC results indicated that the composite material preferentially removed the lower molecular weight components and TOC removal data fitted well to the pseudo-second order. The results of the SEC, SEM, Bet surface area, FTIR analysis and kinetics models showed that the removal mechanism by PAn + Bei composite material was the ion exchange process.

### References


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