REMOVAL OF SULFATE FROM WATER AND WASTEWATER BY SURFACTANT-MODIFIED COIR PITH, AN AGRICULTURAL SOLID ‘WASTE’ BY ADSORPTION METHODOLOGY

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ABSTRACT

The surface of coconut coir pith, a lignocellulosic polymer was modified with a cationic surfactant, hexadecyltrimethylammonium bromide and used as an adsorbent for the removal of sulfate from aqueous solutions. Optimum pH for maximum removal of sulfate was found to be 2.0. Adsorption has been found to be concentration dependent and follows pseudo second order kinetics. Langmuir, Freundlich and Dubinin-Radushkevich isotherms were applied to describe the equilibrium data and the system followed all the three isotherms and the Langmuir adsorption capacity of the adsorbent was found to be 8.8 mg g\(^{-1}\). Desorption studies showed that recovery of sulfate from the spent adsorbent was feasible. Effect of foreign anions on the adsorption of sulfate was also examined. Removal of sulfate by surfactant modified coir pith from industrial wastewater was also tested.

INTRODUCTION

Numerous applications of coconut coir pith, an agricultural solid waste obtained during the separation of fiber from coconut husk, arise from their properties of high porosity and adsorption capacities. It is estimated that the production of coir pith in India is about 7.5 million tons per year [1]. Raw coir pith consists of 35.0% cellulose, 25.2% lignin, 7.5% pentosans, 1.8% fats and resins, 8.7% ash content, 11.9% moisture content and 10.6% other substances [2]. α cellulose and lignin are important sources of relatively easily accessible hydroxyl units that can be used for the attachment of several functional groups [3]. Coir pith shows good adsorption towards dyes [4]. The availability of coir pith in large quantities as solid waste has stimulated further research for new applications. One such new field is anion adsorption. Raw coir pith does not show any affinity for anions without prior modification. In order to enhance its sorption capabilities towards anions, the surface of coir pith was modified using a cationic surfactant, hexadecyltrimethylammonium (HDTMA) bromide. After modification, coir pith exhibits high sorption capacity for anionic contaminants [5,6].

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Sulfate is a common constituent of many natural waters and wastewaters, and is sometimes present in high concentrations. Industrial wastewaters are responsible for most anthropogenic emissions. Certain industrial effluents may contain several thousands of mg L\(^{-1}\) domestic sewage contains typically less than 500 mg L\(^{-1}\) [7]. The damage caused by sulfate emissions is not direct, since sulfate is a non-toxic compound. However, high sulfate concentrations can unbalance the natural sulfur cycle [8]. Several processes can be applied to promote the removal of dissolved sulfate. Chemical precipitation through the addition of barium or calcium salts is an alternative, mainly applied to the treatment of wastewaters that contain high sulfate concentrations. Using membranes is another alternative. However, the relatively high cost and energy consumption, proportional to the sulfate concentration, should be taken into account. Adsorption is an effective and economical method to treat sulfate-containing wastewaters. So this paper examines the sorption of sulfate present in aqueous solution using surfactant-modified coir pith as adsorbent and its applicability is also extended to fertilizer industry wastewater. Effects of contact time, adsorbent dose, pH and temperature on the removal process have been investigated.
MATERIALS AND METHODS

The experimental solutions of sulfate were prepared using analytical grade sodium sulfate, obtained from s.d. Fine Chemicals, Mumbai, India. All reagents used are of analytical grade chemicals and were obtained from Merck and Loba Chemie.

1. Preparation and Characterization of Surfactant-modified Coir Pith (CPHDHTMA)

Waste coir pith was collected from local coconut coir industries and dried in sunlight. Then it was ground and sieved to obtain the particle size of 150-250 μm. The surfactant modified coir pith was prepared as follows: coir pith and 2% HDTMA solution was taken in a 500 mL conical flask and agitated at 200 rpm on a shaker machine for 5 h. Then the suspension was left undisturbed to separate the liquid and the coir pith. The liquid was discarded and the modified coir pith was washed with distilled water several times to remove superficially held surfactant. The modified coir pith was dried in a hot air oven at 60 °C for 8 h. Then it was characterized by Fourier transform infrared (FTIR) spectra, CHN analysis and surface area analysis. Leaching of the surfactant was studied by agitating 0.5 g of the adsorbent with 50 mL of distilled water, at different pH values for 2 h. After agitation, the supernatant was discarded and the adsorbent was filtered using Whatman filter paper and washed gently with water to remove any superficially held surfactant. Then it was dried in hot air oven and analyzed for C, H and N using CHN analyzer (Vario EL III, Germany).

pH measurements were made using a pH meter (Elico, model LI-107, Hyderabad, India). The surface area was measured by a Quantasorb model surface area analyzer. The IR spectra of the samples were recorded on a Shimadzu FTIR spectrophotometer. Orbital shaker machine (Scientific Systems, Chennai, India) was used for batch adsorption studies.

2. Batch Mode Adsorption Studies

Batch mode adsorption studies were carried out by shaking 100 mL conical flasks containing 0.20 g of CPHDTMA and 50 mL of sulfate solution of desired concentration on an orbital shaker machine at 160 rpm, 32 °C and at an initial pH 2.0. The solution pH was adjusted with 0.1 M HCl and 0.1 M NaOH solutions. At the end of the adsorption period the supernatant was separated by centrifugation at 3000 rpm for 30 min. Then the concentration of the residual sulfate ion was determined. The amount of sulfate adsorbed was calculated from the concentration in the solution before and after adsorption. Effect of pH on the adsorption of sulfate onto CPHDTMA was studied for the sulfate concentrations of 20 and 40 mg L⁻¹. Effect of contact time was studied by withdrawing the samples from the shaker at predetermined time intervals and residual sulfate concentration was analyzed as above and bromide ion release from the adsorbent surface was also analyzed simultaneously by phenol red method [9]. Effect of adsorbent dose was studied with different adsorbent doses (100-1000 mg 50 mL⁻¹) for the sulfate concentrations 10-50 mg L⁻¹.

2.1 Effect of foreign ions

Effect of Mo(VI), Cr(VI), PO₄³⁻, SCN⁻, Cl⁻, V(V) and NO₃⁻ at various concentrations on adsorption of sulfate by CPHDTMA was studied.

2.2 Temperature studies

Effect of temperature on adsorption of sulfate was studied using 10-50 mg L⁻¹ sulfate and 0.20 g of the adsorbent at 32, 40, 50 and 60 °C in the thermostated rotary shaker machine.

2.3 Batch mode desorption studies

The adsorbent used for the adsorption of 20 and 40 mg L⁻¹ of sulfate was separated from the solution by suction-filtration using Whatman filter paper and washed gently with water to remove unadsorbed sulfate. Several such samples were prepared. Batch desorption experiments were conducted as a function of desorbing solution pH. To each 100 mL conical flask, 0.2 g of sulfate loaded adsorbent and 50 mL of desorbing solution at pH 2.0-11.0 (adjusted using 1 M HCl/NaOH) were added and agitated for equilibrium time. Then the desorbed sulfate was estimated as before.

RESULTS AND DISCUSSION

1. Characterization of the Adsorbent

The percentages of C, H and N present in the modified and unmodified coir pith are presented in Table 1. The increase in the C, H and N content is due to the adsorption of HDTMA onto coir pith surface. It was found that 198 mg g⁻¹ of HDTMA was adsorbed on coir pith surface. The Brunauer Emmett Teller (BET) surface area of CPHDTMA (1.9 m² g⁻¹) was lower than that of unmodified coir pith (2.3 m² g⁻¹). Sorption of HDTMA on coir pith caused a decrease in the surface area relative to the unmodified coir pith. Apparently, the adsorption process led to a constriction of the pore channels, as a result of attachment of the surfactant moieties to the internal framework surfaces. Similar reduction in the BET surface area was
Table 1. Elemental composition of (a) raw coir pith, (b) CPHDTMA and (c) CPHDTMA after leaching at different pH values

<table>
<thead>
<tr>
<th>Sample</th>
<th>C%</th>
<th>H%</th>
<th>N%</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a) Raw coir pith</td>
<td>40.68</td>
<td>5.13</td>
<td>0.44</td>
</tr>
<tr>
<td>(b) CPHDTMA</td>
<td>53.21</td>
<td>7.31</td>
<td>1.23</td>
</tr>
<tr>
<td>(c) CPHDTMA after leaching at</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(i) pH = 2.0</td>
<td>53.17</td>
<td>7.36</td>
<td>1.18</td>
</tr>
<tr>
<td>(ii) pH = 7.0</td>
<td>53.61</td>
<td>7.49</td>
<td>1.20</td>
</tr>
<tr>
<td>(iii) pH = 11.0</td>
<td>53.22</td>
<td>7.46</td>
<td>1.24</td>
</tr>
</tbody>
</table>

observed during the modification of montmorillonite with HDTMA [10]. The FTIR spectra of raw coir pith, CPHDTMA and sulfate loaded CPHDTMA are shown in Fig. 1. The region below 1550 cm\(^{-1}\) is the fingerprint region and the absorption cannot be assigned clearly to any particular vibration because it corresponds to complex interacting vibration systems. The region between 1800 and 3500 cm\(^{-1}\) presents two major absorption bands centered at 3420 cm\(^{-1}\) (hydrogen bonded OH group) and 2921 cm\(^{-1}\) (C-H stretching of CH\(_2\) group). The intensity of the band at 2921 cm\(^{-1}\) increased due to the increase in the aliphatic carbon content in CPHDTMA, which in turn is due to the adsorption of HDTMA on to coir pith surface. No change in the spectrum was observed for the sulfate loaded CPHDTMA.

Sorption of cationic surfactant from solution onto solid surfaces has undergone extensive study in recent years. Several mechanisms were attributed to the sorption of cationic surfactants onto solid surfaces [11]. The non polar (alkyl) portion of HDTMA most likely interacts with coir pith surface through hydrophobic bonding and the polar (positively) charged head groups pointed towards the bulk of the solution, making the surface potential positive. In solution, certain groups in the pith, such as lignin and cellulose, lose hydrogen ions and form a particle surface with a negative potential [12]. Another possible mechanism is the electrostatic attraction of surfactant cations on the coir pith surface, which offers a negative potential. As the concentration of surfactant is well above the critical micelle concentration [13], adsorption increases through hydrophobic interaction between hydrocarbon chains of the surfactant in solution and the surfactant molecules already adsorbed on the surface. In both mechanisms, the surface gets a positive potential, which is conducive for the removal of anionic contaminants.

2. Effect of pH

Maximum removal was found at pH 2.0 (Fig. 2). As the pH was increased removal decreased and was less than 5% at pH 11.0. At pH 2.0, degree of surface protonation was high and so surface offers maximum positive charge for the adsorption of sulfate. In acidic solutions, sulfate anions can be exchanged with the counteranions on the protonated amine sites. As the pH was increased, the degree of surface protonation decreased and the removal also decreased. Above pH\(_{ZPC}\) (9.1), the removal was negligible because of the reversal of the surface charge. So the subsequent experiments were carried out at initial pH, 2.0.

3. Effect of Contact Time and Initial Sulfate Concentration

Figure 3 shows that the uptake of sulfate (mg g\(^{-1}\)) increased with increase in sulfate concentration and remained nearly constant after equilibrium time. The equilibrium time was found to be 30, 50, 80, 90 and 100 min for 10, 20, 30, 40 and 50 mg L\(^{-1}\) of sulfate, respectively. The amount of sulfate adsorbed (q\(_e\)) increased from 2.3 to 7.6 mg g\(^{-1}\) as the concentration was increased from 10 to 50 mg L\(^{-1}\). It also showed that the adsorption was rapid in the initial stages and
Table 2. Comparison of first order and second order adsorption rate constant and calculated and experimental $q_e$ values for different initial sulfate concentrations

<table>
<thead>
<tr>
<th>Initial sulfate conc (mg L$^{-1}$)*</th>
<th>$q_e$ (exp.) (mg g$^{-1}$)</th>
<th>$k_1$ (min$^{-1}$)</th>
<th>$q_e$ (calc.) (mg g$^{-1}$)</th>
<th>$R^2$</th>
<th>$k_2 \times 10^3$ (g mg$^{-1}$ min$^{-1}$)</th>
<th>$q_e$ (calc.) (mg g$^{-1}$)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>2.27</td>
<td>0.1112</td>
<td>2.59</td>
<td>0.9844</td>
<td>0.0240</td>
<td>3.23</td>
<td>0.9993</td>
</tr>
<tr>
<td>20</td>
<td>4.14</td>
<td>0.0751</td>
<td>3.77</td>
<td>0.9985</td>
<td>0.0185</td>
<td>5.03</td>
<td>0.9990</td>
</tr>
<tr>
<td>30</td>
<td>5.81</td>
<td>0.0422</td>
<td>4.02</td>
<td>0.9693</td>
<td>0.0162</td>
<td>6.31</td>
<td>0.9985</td>
</tr>
<tr>
<td>40</td>
<td>6.88</td>
<td>0.0355</td>
<td>4.67</td>
<td>0.9801</td>
<td>0.0126</td>
<td>7.40</td>
<td>0.9972</td>
</tr>
<tr>
<td>50</td>
<td>7.57</td>
<td>0.0301</td>
<td>4.96</td>
<td>0.9820</td>
<td>0.0111</td>
<td>8.01</td>
<td>0.9957</td>
</tr>
</tbody>
</table>

*Conditions: Temperature, 32 °C; Adsorbent dose, 0.2 g 50 mL$^{-1}$; pH 2.0.

4. Adsorption Kinetics

Adsortion kinetic data of sulfate are analyzed using the Langergren first order rate equation [14]:

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

(1)

where $q_e$ and $q_t$ are the amounts of sulfate adsorbed (mg g$^{-1}$) at equilibrium and at time $t$ (min), respectively, and $k_1$ is the Langergren rate constant of first order adsorption (min$^{-1}$). Values of $q_e$ and $k_1$ were calculated from the plots of log ($q_e$ vs) vs $t$ (not shown). The second order kinetic model [15] can be represented as:

$$\frac{t}{q_t} = k_2 q_e^2 + \frac{t}{q_e}$$

(2)

where $k_2$ is the rate constant of second order adsorption (g mg$^{-1}$ min$^{-1}$). Values of $k_2$ and $q_e$ were calculated from the plots of $t/q$ vs $t$ (Fig. 4).

Table 2 showed that the experimental $q_e$ values were closer to the calculated $q_e$ values obtained from the second order kinetic plots except for 10 mg L$^{-1}$ concentration compared to those of the first order kinetic plots and correlation coefficients for the second order kinetic plots were above 0.99. These results indicate that the adsorption process generally follows the second order kinetic model. Similar results have been observed in the adsorption of sulfate onto ZnCl$_2$ activated coir pith carbon [16] and adsorption of phosphate onto CPHDTMA [5].

5. Adsorption Isotherms

The equilibrium adsorption isotherm is fundamental in describing the interactive behavior between adsorbate and adsorbent, and is important in the design of adsorption systems. Generally used Langmuir isotherm is represented by the following equation [17]:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0}$$

(3)
Table 3. Langmuir, Freundlich and Dubinin-Radushkevich constants

<table>
<thead>
<tr>
<th></th>
<th>Langmuir</th>
<th>Freundlich</th>
<th>Dubinin-Radushkevich</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Q_0$</td>
<td>(mg g$^{-1}$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8.76</td>
<td>0.3047</td>
<td>0.9968</td>
<td></td>
</tr>
<tr>
<td>$b$</td>
<td>(L mg$^{-1}$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3.60</td>
<td>3.15</td>
<td>0.9909</td>
<td></td>
</tr>
<tr>
<td>$R^2$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.9968</td>
<td>0.9818</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$k_l$</td>
<td>(mg$^{-1}$L$^{1/n}$g$^{-1}$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.45</td>
<td>2.47</td>
<td>0.9818</td>
<td></td>
</tr>
<tr>
<td>$n$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.47</td>
<td>0.9818</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

where $C_e$ is the concentration of sulfate solution (mg L$^{-1}$) at equilibrium. The constant $Q_0$ signifies the adsorption capacity (mg g$^{-1}$) and $b$ is related to the energy of adsorption (L mg$^{-1}$). Langmuir isotherm with the experimental data is shown in Fig. 5. Values of $Q_0$ and $b$ were calculated from the slope and intercept of the linear plot of $C_e/q_e$ vs $C_e$ and are presented in Table 3.

The essential characteristics of Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter, $R_L$ [18].

$$ R_L = \frac{1}{1 + b C_0} $$

where $b$ is the Langmuir constant (L mg$^{-1}$) and $C_0$ is the initial sulfate concentration (mg L$^{-1}$). $R_L$ values indicate the type of isotherm. $R_L$ values for sulfate sorption decreases with increase of concentrations of sulfate and is shown in Fig. 6, which indicates that sorption is more favorable for the higher initial concentrations than for the lower ones [19]. The $R_L$ values between zero and one indicate a favorable adsorption process. Freundlich isotherm was also applied for the adsorption of sulfate [20].

$$ \log q_e = \log k_f + \frac{1}{n} \log C_e $$

where $k_f$ and $n$ are constants incorporating all factors affecting the adsorption process (adsorption capacity and intensity of adsorption). Freundlich isotherm with the experimental data is shown in Fig. 5. Values of $k_f$ and $n$ were calculated from the intercept and slope of the linear plot of $\log_{10} (q_e)$ vs $\log_{10} C_e$ and are presented in Table 3. D-R isotherm is more general than the Langmuir isotherm because it does not assume a homogeneous surface or constant adsorption potential. The linear form of D-R isotherm [2] is

$$ \ln q_e = \ln q_m - B \varepsilon $$

where $B$ is a constant related to the mean free energy of adsorption per mol of the adsorbate (mol$^2$ J$^{-2}$), $q_m$ is the theoretical saturation capacity (mol g$^{-1}$) and $\varepsilon$ is the Polanyi potential, which is equal to $RT \ln(1+1/C_e)$, where $R$ (J mol$^{-1}$ K$^{-1}$) is the gas constant and $T$ (K) is the absolute temperature. Hence by plotting $\ln q_e$ versus $\varepsilon$, it is possible to obtain the value of $q_m$ from the intercept and the value of $B$ from the slope. Values of $q_m$ and $B$ are presented in Table 3. The constant $B$ gives an idea about the mean free energy $E$ (kJ mol$^{-1}$) of adsorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinity in the solution and can be calculated from the relationship [21]

$$ E = \frac{1}{\sqrt{2B}} $$

If the magnitude of $E$ is between 8 and 16 kJ mol$^{-1}$, the adsorption process follows ion exchange, while for the values of $E$ lesser than 8 kJ mol$^{-1}$, the adsorption process is of physical nature. High values of $E$ of 24.7 ± 3.2 kJ mol$^{-1}$ show that strong chemical bond formation between adsorbate and adsorbent [22]. Since the value of $E$ is 12.6 kJ mol$^{-1}$, the adsorption follows ion
exchange mechanism.

All the three isotherms showed good fit to the experimental data (Fig. 5). The adsorption capacity of D-R isotherm is higher compared to Langmuir isotherm. This difference in adsorption capacity can be interpreted in terms of the assumptions taken into consideration while deriving these adsorption models [22].

6. Effect of Adsorbent Dose and Temperature

As the adsorbent dose increased, the per cent removal of sulfate was also increased. This is due to the increase of number of sites for adsorption of sulfate adsorption. Quantitative removal of sulfate was achieved by increasing the dose to 1.0 g.

There was no significant increase in the amount adsorbed \([q_e \text{ (exp)}]\) was around 9 mg g\(^{-1}\) with increase in temperature.

The change in standard free energy \((\Delta G^0)\) of adsorption was calculated using the following equation:

\[
\Delta G^0 = -RT \ln b \tag{8}
\]

where \(R\) is the gas constant (J K\(^{-1}\) mol\(^{-1}\)) and \(b\) is the Langmuir constant (L mg\(^{-1}\)) and \(T\) is the temperature in K. \(\Delta G^0\) was found to be -25.6 kJ mol\(^{-1}\) at 32 °C. The negative value of \(\Delta G^0\) at 32 °C indicates that the adsorption process is spontaneous.

7. Effect of Foreign Ions

Chromate, molybdate and thiocyanate affected the removal of sulfate at all concentrations studied (Table 4) Vanadate affected the removal process at higher concentrations of 50 and 100 mg L\(^{-1}\). This shows that the above foreign ions compete for the adsorption sites with sulfate species and hence there are no specific or exclusive sites for sulfate ions on the surface of the adsorbent. This also supports ion exchange mechanism of sulfate uptake. Other anions such as PO\(_4\)\(^{3-}\), Cl\(^{-}\) and NO\(_3\)\(^{-}\) did not affect the adsorption of sulfate in the concentration range of 10-100 mg L\(^{-1}\).

8. Desorption Studies

Since sulfate ion and sorbent interaction is strong at pH 2.0 to 5.0, less desorption was observed at this pH range. On the other hand above this pH range, the affinity for metal ion is weaker and there was sulfate release. Above pH 5, the desorption increased progressively. As the desorbing pH was increased from 5.0 to 11.0, the per cent desorption of sulfate increased (Fig. 2). Desorption of sulfate is due to the displacement of sulfate ions from the adsorbent sites by OH\(^{-}\) ions. So the adsorption occurs mostly by ion exchange mechanism. Very high desorption of sulfate from the spent adsorbent makes the removal process economical because both adsorbent and sulfate ions are regenerated and are recyclable.

**TEST WITH INDUSTRIAL WASTEWATER**

The effectiveness of CPHDTMA was tested by extending its applicability for the treatment of fertilizer industry wastewater containing sulfate collected from a fertilizer industry situated at Coimbatore, India. The wastewater contains SO\(_4\)\(^{2-}\) (35 mg L\(^{-1}\)), PO\(_4\)\(^{3-}\) (22 mg L\(^{-1}\)), NO\(_3\)\(^{-}\) (58 mg L\(^{-1}\)), Ca\(^{2+}\) (42 mg L\(^{-1}\)), Mg\(^{2+}\) (11 mg L\(^{-1}\)) and Na\(^{+}\) (23 mg L\(^{-1}\)). Effect of pH on the adsorption of sulfate from the wastewater was studied by using 0.2 g 50 mL\(^{-1}\) of CPHDTMA and the effect of adsorbent dose on the removal was studied using the adsorbent dose varied between 0.1 to 1.5 g 50 mL\(^{-1}\). Results showed that the effect of pH on the removal of sulfate from fertilizer industry wastewater followed the same trend as that of the pure solution. But the per cent removal decreased. Effect of adsorbent dose showed that the removal of sulfate from the fertilizer industry wastewater increased with increase in dose. Dose required for the maximum removal of sulfate increased for the wastewater. This may be attributed to the fact that other ions present in the wastewater inhibit the adsorption of sulfate.

**CONCLUSIONS**

HDTMA bromide was used to modify the surface of the lignocellulosic polymer, coir pith. Removal of sulfate ions from aqueous solution by surfactant-modified coir pith was found to be effective. Optimum pH for sulfate removal was found to be pH 2.0. Equilibrium adsorption data showed good fit to Langmuir, Freundlich and D-R isotherms. Langmuir adsorption capacity was found to be 8.8 mg g\(^{-1}\). Adsorption process followed second order kinetic model. Thermodynamical studies indicated that the adsorption process was spontaneous. Desorption of Br\(^{-}\) from the adsorbent surface during the adsorption of sulfate and desorption studies showed that ion exchange was the
major mechanism governing the uptake of sulfate. Very high desorption of sulfate from the spent adsorbent regenerates both the adsorbent and sulfate, making the process economically viable. Quantitative removal of sulfate from fertilizer industry wastewater was also achieved using CPHD TMA.

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REFERENCES


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