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Metal etching industry wastewater pretreatment by coagulation and ion exchange resins

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ABSTRACT

The removal of dissolved organic compounds (DOC) in the metal etching industry wastewater was tested by ion-exchange resins (IERs), coagulation and their combination. The appropriate IER was selected by batch and column tests out of Amberlite IR-120, IRA-900 and XAD-4. XAD-4 was the most efficient IER for the DOC removal in addition to removing the added surfactant, showing that the DOC was mostly hydrophobic in nature. The XAD-4 column treated 250 bed volumes. The minimum effluent total organic carbon (TOC) was 30 mg/L during first 50 BV and gradually increased to 90 mg/L at 250 BV. It showed that a column with 0.1 m^3 bed volume would be required for the influent flow rate of 24 m^3/ day, with daily regeneration. The coagulation with iron salt removed hydrophilic DOC and the minimum and optimum coagulant doses for DOC removal were 2.1 and 15.5 mg Fe(III)/mg TOC, respectively. A combination of coagulation and XAD-4 adsorption decreased minimum effluent TOC to 25 mg/L and 7 mg/L after 2.1 and 15.5 mg Fe(III)/mg TOC pretreatments, respectively. The bed life of XAD-4 was not increased because adsorption capacity is a specific property. Combination process can be used as pretreatment before RO process.

Keywords: Metal etching industry; Wastewater recycle; Coagulation; Ion exchange; Hydrophobicity/hydrophilicity

1. Introduction

Water resources are decreasing day by day with the increasing human population and industrialization. Generation of new water resources is impossible therefore only option is to reuse the wastewater for domestic, industrial, and agricultural purposes. Reverse osmosis (RO) is the best option to treat wastewater for reuse [1]. Membrane fouling is a major problem of RO process. Biofouling can be a major issue when the influent of RO contains high concentration of dissolved organic compounds (DOC). Microbes grow on the membrane consuming the DOC in the influent water and cause the biofouling. The DOC should be removed to a level to minimize the microbial growth on the RO membranes.

The conventional biological systems require technical knowledge and space. Adsorption is effective and easy to use process for DOC removal and many adsorbents are tested [2–4]. Granular-activated carbon (GAC) is a conventional material used for the DOC removal. The regeneration of GAC is difficult and expensive due to strong adsorption-desorption

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hysteresis with the pore deformation [5]. The polymeric adsorbents and ion exchange resins (IERs) are attractive adsorbents due to their diverse structure and easy regeneration [6–8]. Polymeric adsorbents have a wider range of ionic types. Amberlite XAD-4, IR-120, and IRA-900 are nonionic polymeric adsorbent, cationic and anionic IERs, respectively. Amberlite XAD-4 is a nonionic crosslinked polymer with macroreticular structure (containing both a continuous polymer phase and a continuous pore phase) and the aromatic nature of its surface. Amberlite IRA-900 resin is a macroreticular polystyrene strong base anion-exchange resin containing quaternary ammonium groups. Amberlite IR120 resin is a gel type strongly acidic cation-exchange resin of the sulfonated polystyrene type.

The Amberlite XAD-4 is reported to be one of the best polymeric adsorbents for removing DOC from wastewater [8,9]. XAD-4 is recommended for the adsorption of compounds of molecular weight less than 1,000 with aromatic character and a high local electron density due to the presence of nitrogen, sulfur, chloride, or bromide. With a dipole moment of 0.3 (data from the manufacturer), XAD-4 can adsorb a wide variety of compounds from wastewater. Therefore this study was planned to test the XAD-4 as an alternative of GAC. The coagulation with the ferric ions is reported to reduce the DOC from the water. This lower DOC in wastewater facilitates the RO process by reducing the fouling [10].

In the metal-etching process, metal surface is coated with "ground" that may be asphalt, bitumen or special kind of wax. To expose, the required area of metal surface ground is scratched. During etching process, scratched area is exposed to biting solution such as nitric acid. After the etching process, the solvents and surfactants are used to remove the coating from the plates. These all processes involve different kinds of organic compounds that ultimately add up in the wastewater giving a high DOC of complex type. These types of DOCs in high concentrations are difficult to remove by a single process. Wastewater with high DOC is difficult to treat with RO for reuse purposes. Advanced pretreatment processes should be tested for RO process. The metal etching industry's wastewater has different types of organic compounds in it; therefore, a combination of processes can be used to increase the overall process efficiency [11].

The comparative adsorption of XAD-4 and GAC has been reported and found that XAD-4 has less adsorption capacity [5]. Adsorption and coagulation have been tested individually as well as in combination [12–17], but no one has tested the combination of coagulation with XAD-4 as an alternative to activated carbon. Some authors tested the magnetic ion-exchange resin as pretreatment before alum coagulation, although it did not improve the results [18,19]. Some authors have tested the enhanced adsorption using the modified materials [8,20], although pretreatment can be a choice despite adsorbent modification. It has already been reported that aromatic polymer adsorbent like XAD-4 are more efficient than aliphatic adsorbents like XAD-8, but these are studied for individual compounds in water [21]. The pretreatment with coagulation followed by IERs has been studied by some authors but they have used only cationic or anionic resins without reporting the selection of appropriate IER [10,22]. In this study, the wastewater is different from natural water being a mixture of different types of organics in high concentration that are difficult to remove by a single process. Therefore, we first selected the appropriate IERs/adsorbent that was a factor of charge type of organics and then continued the next study. These processes were tested in the batch and continuous columns to verify the efficiency. In the previous studies, the coagulation is reported to treat the natural water and domestic wastewater for removal of the natural organic matter that is hydrophobic macromolecular [23,24].

This study was focused at the pretreatment of industrial wastewater aimed to reuse after RO process. This wastewater contained a wide variety of organic compounds with low molecular weights. To remove the high concentration of DOC tested the IER and polymeric adsorbent as they are easier to regenerate than GAC. First of all appropriate IER and polymeric adsorbent was selected depending on the DOC being hydrophobic or ionic in nature. Coagulation was tested for DOC removal and the results were different from the reported literatures that had focused on the removal of natural organic matter from natural waters. A combination of adsorption on IERs/ polymeric adsorbent and coagulation were tested as expected to increase the DOC removal due to selective DOC removal in the two processes.

2. Experimental

2.1. Material

The wastewater from the metal-etching industry was taken in plastic containers and kept at 4˚C till further use. Before use, the wastewater was filtered with 0.45-μm membrane to filter the suspended particles. The IERs Amberlite IRA-900 and IR-120 were from Sigma Aldrich. Amberlite IRA-900 was a macroreticular, polystyrene, type one, strong base, anion-exchange resin containing quaternary ammonium groups. Amberlite 120 is a gel type strongly cationic-exchange resin of the sulfonated polystyrene type. Amberlite XAD-4 was from Sigma Aldrich. Amberlite XAD-4 is a polymeric adsorbent in the form of white insoluble beads. It is a nonionic crosslinked polymer that derives its adsorptive properties from its macroreticular structure. These adsorbents were cleaned before use. XAD-4 was regenerated with methanol and rinsed with DI water before use. IR-120 was regenerated with 1 M HCl and 1 M NaCl and rinsed with DI water. IRA-900 was kept in 1 M NaOH for five days and replaced the new 1 M NaOH solution with fresh one daily and then rinsed extensively with de-ionized water. The other chemicals used in this study were of analytical grade and used without any purification.

2.2. Experimental systems

2.2.1. IERs/polymeric adsorbent selection and kinetics

The IERs/polymeric adsorbent were taken in 500-ml bottles, each 10 g. Added 500 ml raw wastewater (RWW) to the bottle, purged with N_2 gas and kept in the shaking incubator for shaking at 150 rmp and 20℃. Samples were collected at predefined time intervals to test the kinetics of TOC removal by the IERs/polymeric adsorbent.

2.2.2. Continuous column tests with IERs/polymeric adsorbent

Continuous adsorption tests were run with XAD-4, IR-120, and IRA-900 columns. The wastewater was treated with all IERs/polymeric adsorbent in column test to select the IER/polymeric adsorbent with maximum efficiency for further study of wastewater treatment. The column dimensions were as: column diameter 11 mm and bed depth 16 cm giving bed volume of 15 ml. The flow rate was kept at 3.7 ml/min to get the empty bed contact time (EBCT) of 4 min. First 10 bed volumes (BVs) were wasted and then took the samples for TOC analysis. New column setup was installed for the optimization of the column efficiency with the same column dimensions. The flow rate was varied to get the EBCT of 1.6, 3.0, 4.0, 7.1, and 15.0 min and took samples for TOC test.

This wastewater contained surfactants in it (the information provided by the metal etching industry). The sample of surfactant present in wastewater was taken from the industry and a solution of the surfactant was prepared in de-ionized water to get a TOC of 100 mg/L. This solution was treated with columns of IERs/polymeric adsorbent of different ionic types with above column dimensions and 4 min EBCT.

The XAD-4 was selected for the treatment of wastewater under study. The breakthrough tests with XAD-4 were performed to quantify the capacity of XAD-4 to treat the wastewater in the field with above given column dimensions and 4 min EBCT. During this breakthrough test, for four BVs only surfactant solution was added as influent and tested the effluent for TOC. The same breakthrough test was repeated without using surfactant solution as influent.

2.2.3. Coagulation test

Coagulation testes with ferric chloride were performed in 1L beaker. Pre-weighed ferric chloride was added to different beakers containing 500 ml wastewater, changed the pH to 6.2 and stirred for five minutes at 80 rpm and for 30 min at 20 rpm. Let the flocks settle and filtered the supernatant with 0.45-μm membrane. The coagulant quantities for optimum and maximum TOC removals were selected as pretreatments for GAC tests based on TOC removal. The wastewater treated by the selected coagulant doses was characterized by the IERs for the charge type of DOCs [23]. Selected coagulation pretreatments for XAD-4 tests were performed in batches of 4L in glass beakers (5L).

2.2.4. Combination of coagulation and XAD-4 adsorption

In the final test the wastewater treated by the coagulation was treated by XAD-4 column to optimize the DOC removal. The adsorption test was performed with RWW and wastewater treated with selected coagulant doses. The pH was changed to 7.0 before column test. The column specs were: diameter 11 mm, bed depth 16 cm, bed volume 15 ml, and EBCT 4 min. The samples were collected and analyzed for TOC.

TOC was analyzed by the TOC analyzer TOC-VCPH, Shimadzu.

3. Results and discussion

3.1. Ion exchange resin treatment

3.1.1. Kinetics in batch tests

The metal-etching industry's wastewater tested in this study had 100 mg/L TOC (Table 1). The adsorption kinetics of DOC on IERs was found to be very fast (Fig. 1). The equilibrium was reached within three hour of mixing. From the batch test, it could be concluded that XAD-4 was most efficient for the DOC removal with maximum of 64% DOC removal.

Table 1
Characteristics of raw wastewater

Parameter	RWW (average ± SD)
pH	6.1
Conductivity (μS/cm)	535
TS (mg/L)	501 ± 0.5
TDS (mg/L)	488 ± 1.5
FDS (mg/L)	438 ± 1.2
COD_{Cr} (mg/L)	218 ± 1
TOC (mg/L)	101 ± 0.3

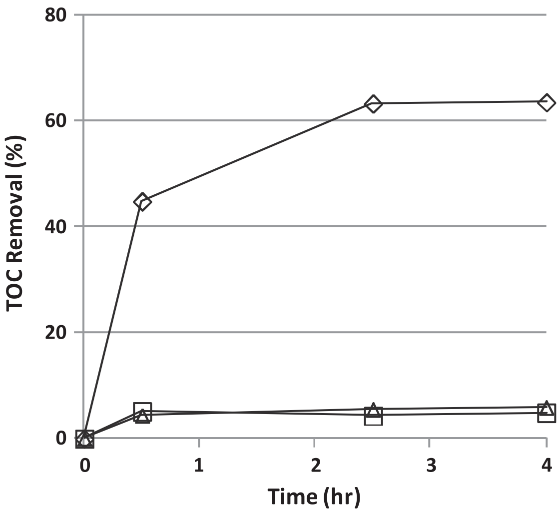

Fig. 1. TOC removal kinetics with different ion exchange resins, Δ—IR-900, □—IR-120, and ◇—XAD-4.

IRA-900 and IR-120 were less efficient than XAD-4. This indicated that wastewater contained a major fraction of hydrophobic compounds (≈65%), while the hydrophilic fraction of organic compounds was only ≈35%. The removal of highly ionic species by the IRA-900 and IR-120 was 6 and 5%. During a previously published study [11] we found that in this wastewater the molecular weights of the organics ranged between 146 and 453 as detected by HPLC/MS/MS. There was no correlation between molecular weight and hydrophobicity of organics. Macromolecules like humic acid and fulvic acid were not found in the wastewater. The organics in this wastewater were of synthetic origin used during coating and washing of metal plates. Therefore, these organics behaved differently from the natural organic matter in natural waters as was claimed by different authors for natural waters especially with the anionic IERs [18,19].

3.1.2. Column test with different IERs/polymeric adsorbent

The TOC removal efficiencies were compared in a continuous process to finalize the selection of more relevant IERs for wastewater treatment. The wastewater was treated by the XAD-4, IR-120, and IRA-900 being nonionic polymeric adsorbent, and cationic and anionic IERs, respectively, in a continuous column test. XAD-4 was the most efficient adsorbent for the TOC removal (Fig. 2). The IERs IR-120 and IRA-900 were less efficient with removal efficiency of 9 and 5%, respectively. This indicated that the wastewater had little quantity of highly cationic and anionic organic compounds. As this wastewater was different from the natural water and had organic compounds of synthetic origin. HPLC/MS/MS showed that the compounds were of low molecular weight, but their

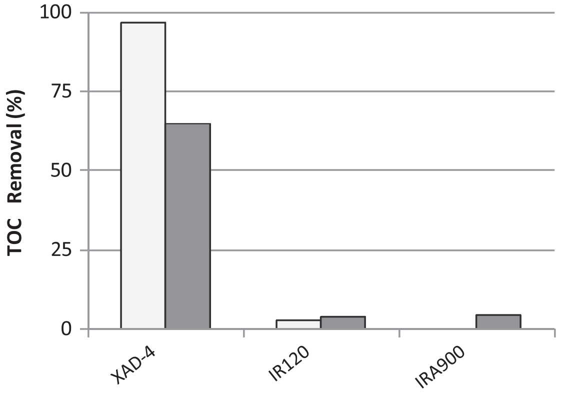

Fig. 2. TOC removal from RWW and surfactant solution with different ion exchange resins, blank bars—surfactant solution, filled bars—raw wastewater.

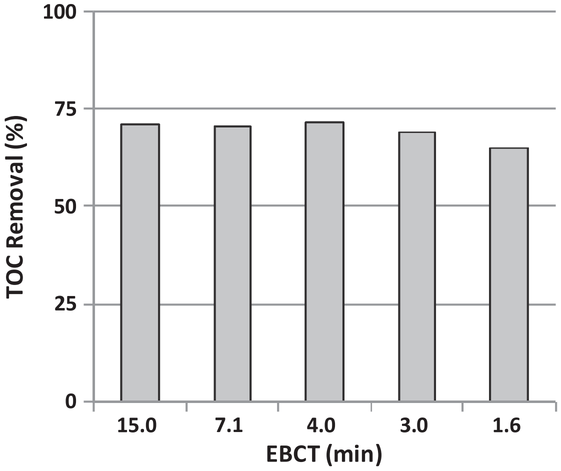

Fig. 3. TOC removal with variation of EBCT for XAD-4 ion exchange resins column.

removal by XAD-4 showed that most of them were hydrophobic in nature.

With XAD-4, the EBCT was varied from 1.6 to 15.0 min to optimize the process efficiency for the removal of DOC from the RWW. It was found that due to the high adsorption rate of organics on the XAD-4 the variation of EBCT had no major impact on the removal efficiency of the IERs columns (Fig. 3). This was different as was found for the GAC column as GAC has different types of pores and it take longer for the molecule to travel from the exterior of GAC particle to the micro pore where it gets adsorbed. In case of, IERs the pore structures are simple and homogeneous therefore adsorption is faster and almost independent of EBCT. For XAD-4, the optimum TOC removal efficiency was at 4.0 min EBCT. For the IERs, there was no major variation of TOC removal efficiency with increasing the EBCT from 1.6 to 15.0 min.

3.1.3. Column test with surfactant only

Characterizations of the surfactants require costly equipments like liquid chromatography coupled with mass spectrometer. In this study the process was kept simple to be used in the laboratory and field. Therefore, to know the charge and effect of surfactant, its removal was tested with IERs/polymeric adsorbent. There was no need to know the name of surfactant once its ionic nature is characterized.

The surfactant sample was taken from the industry and tested for removal by column test with XAD-4, IRA-900, and IR-120 without characterizing for the identity of the surfactant. It was found that XAD-4 removed 97% TOC of surfactant solution, while IRA-900 and IR-120 removed 1 and 3% TOC, respectively (Fig. 2). It showed that surfactant might be non-ionic type. XAD-4 was not only most efficient for the DOC removal from the RWW but also completely removed the surfactant in the wastewater. Therefore, in this study, the presence of surfactant in the wastewater would have no adverse effect on DOC removal efficiency.

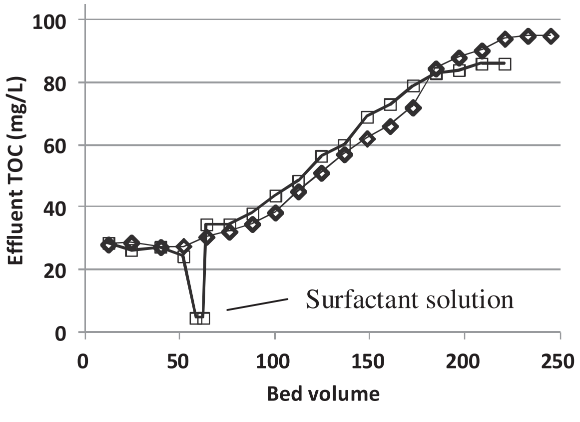

Fig. 4. Breakthrough test of XAD-4 for TOC removal performed in duplicate for RWW, influent - wastewater = 104 mg/L, surfactant solution = 100 mg/L, EBCT = 4 min; bed volume = 15 ml.

3.1.4. Breakthrough test with XAD-4

The breakthrough column test was performed to evaluate the process efficiency in the field test. It was found that the minimum TOC reached was 30 mg/L (Fig. 4). The effluent TOC remained at a minimum only for about 50 BV and then gradually increased to total exhaustion till 250 BV. From the breakthrough parameters, it was found that a column with 0.1 m^3 bed volume would be required for the influent flow rate of 24 m^3/day, with daily regeneration. Though the capital cost is high, but daily regeneration would make the process cost efficient. This test showed that the hydrophilic fraction is the cause of high effluent TOC. A pretreatment step before the XAD-4 column may decrease the effluent TOC near to the RO influent standard by selective removal of hydrophilic fraction.

3.2. Coagulation

Coagulation test was performed with $FeCl_3$ as a coagulant. The dose of $FeCl_3$ was increased to optimize the DOC removal. The TOC removal increased with the increasing Fe(III) dose. Only filtration removed 3% TOC. The minimum effective efficiency was considered to be $\approx$16% removal that was reached with 2.1 mg Fe(III)/mg TOC dose (Fe-2.1). The optimum TOC removal was 34% with 15.5 mg Fe(III)/mg TOC. Therefore, coagulant doses of 2.1 mg Fe(III)/mg TOC (Fe-2.1) and 15.5 mg Fe(III)/mg TOC (Fe-15.5) were selected as minimum effective and optimum effective doses for pretreatments before adsorption treatment. The wastewater treated by Fe-2.1 and Fe-15.5 was analyzed by IERs/polymeric adsorbent for the fractions of DOC [23] (data were not shown here). The analysis with IERs/polymeric adsorbent showed that coagulation removed the hydrophilic fractions preferentially over the hydrophobic fractions. Detailed testing of hydrophilic fraction removed by coagulation using IERs [23] showed that coagulation removed only anionic hydrophilic fraction [11]. The literatures reported that coagulation removed hydrophobic macromolecules. The anomalous behavior in this study was explained on the basis of HPLC/MS/MS data and the charge interaction. HPLC/MS/MS showed that there was no macromolecule in this wastewater and there was no correlation between the size of molecule and hydrophobicity [11]. For this industrial wastewater, organic compounds removed by the coagulation were decided on the basis of charge interaction rather than the size of molecule. During coagulation, ferric chloride created positively charged ferric flocs, for example, ferric dimer $Fe_2(OH)_4^{+2}$ [25]. The interaction between these cationic flocs and anionic hydrophilic fraction removed TOC from the wastewater. In the Fe-2.1 test, only anionic fraction was removed. Whereas with a maximum coagulant dose in the Fe-15.5 test, only 1.1 mg/L of the cationic fraction of DOC that may be due to factors other than charge interaction.

3.3. Combination of coagulation + Ion exchange

Comparative breakthrough tests of XAD-4 were run with the RWW and Fe-2.1 and Fe-15.5 treated wastewaters (Fig. 5). The results of the column run with RWW were quite similar to the test described in "Breakthrough test with XAD-4." The effluent of the column run with Fe-2.1 had lower TOC of 25 mg/L. This was because some of the hydrophilic fraction was removed by the coagulation with iron salt. The column run with Fe-15.5 was most efficient. The minimum effluent TOC was 7 mg/L. It showed that a major part of the hydrophilic fraction had already been removed by the coagulation. The remaining DOC after coagulation was mostly hydrophobic in nature. This remaining DOC was adsorbed by the XAD-4 that is non-ionic polymeric adsorbent. The process could not increase the bed life of XAD-4 column because the adsorption capacity depended on the active sites on the surface of XAD-4 that was a specific property of each XAD-4.

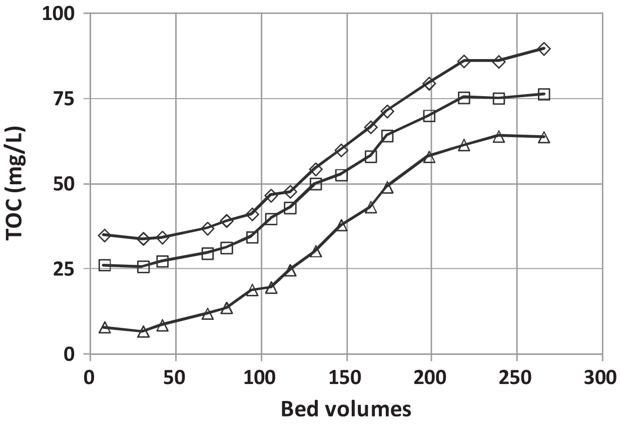

Fig. 5. Effluent TOC of XAD-4 breakthrough test for wastewater, bed volume = 15 ml, EBCT = 4 min, influent TOC were as RWW = 101 mg/L, Fe-2.1 = 82 mg/L, Fe-15.5 = 71 mg/L, ◇—RWW, □—Fe-2.1, Δ—Fe-15.5.

This study indicated that polymeric adsorbent can be a good choice of pretreatment for RO to remove the DOC. In this study the important step was selection of IERs/polymeric adsorbent under batch and continuous modes. IERs/polymeric adsorbent selection was a factor of hydrophobicity/hydrophophilicity and charge type of each compound in the wastewater. In the literature published by different authors, IERs/polymeric adsorbent were randomly selected for natural waters treatment [10,22]. But industrial wastewaters have many types of compounds and selection of appropriate IERs/polymeric adsorbent becomes important during process optimization. In the previously published study GAC treatment after coagulation, we found that GAC preferentially removed the hydrophobic fractions [11]. Regeneration of GAC was difficult and expensive due to strong adsorption-desorption hysteresis with the pore deformation and made it a difficult process. In this study, GAC was replaced by XAD-4 to remove the hydrophobic fractions, as an alternative to GAC after the first step of IER/adsorbent selection. XAD-4 can be easily regenerated reducing the cost of pretreatment before RO process. The pretreatment with coagulation increased the TOC removal efficiency because coagulation predominantly removed the hydrophilic fraction and XAD-4 removed the hydrophobic fractions giving minimum effluent TOC.

4. Conclusions

The pretreatment of metal etching industry wastewater was tested for removing the DOC by coagulation and ion-exchange resins/polymeric adsorbents. Appropriate adsorbent out of Amberlite IR-120, IRA-900, and XAD-4 was selected by the kinetics batch test. The rate of adsorption was very fast and equilibrium reached within 3 h of mixing. The XAD-4 was the most efficient for the DOC removal. The column test run with EBCT of 4 min confirmed the results of batch test. This showed that the major fraction of DOC was hydrophobic in nature, about ≈65%. The surfactant present in the wastewater was removed completely by the XAD-4 resin. The removal of DOC was not very much dependent upon the EBCT because of fast adsorption. Breakthrough test was run with XAD-4 column and minimum effluent was 30 mg/L that increased after 50 BV treatment and treated 250 BV till 90% breakthrough. It showed that a column with 0.1 m^3 bed volume would be required with daily regeneration if the effluent flow rate is

24 m^3/day. During coagulation, increasing the iron salt dose increased total organic carbon TOC removal. Coagulation selectively removed the hydrophilic fraction of DOC. The minimum and optimum coagulant doses selected as pretreatment were 2.1 mg Fe(III)/mg TOC (Fe-2.1) and 15.5 mg Fe(III)/mg TOC (Fe-15.5). Combination of coagulation and XAD-4 adsorption gave minimum effluent TOC of 25 and 7 mg/L for Fe-2.1 and Fe-15.5 pretreatments, respectively. The adsorption capacity is a specific property of the polymeric adsorbent therefore could not be increased. A combination of coagulation and XAD-4 removed hydrophilic and hydrophobic DOC, respectively. This combination of processes can be used to pretreat the wastewater before RO to minimize biofouling.

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