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Investigation of Emulsified Oil Wastewater Treatment with Polymers

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ABSTRACT

Wastewater from metal industry hot milling operations contains oil primarily from coolant sprayed on metal strips to dissipate heat during the rolling of metals. The emulsified oil wastewater for this study was withdrawn from two holding ponds where free oil would rise to the surface and was removed periodically, and used as influent for the chemical addition (CA)–gravity separation (GS) process. The principal objective of this research was to evaluate the effectiveness of different polymer addition systems through a CA–GS process for oil/grease (O/G) and total suspended solids (TSS) removal. Polymers from two corporations were investigated. A dual polymer system was recommended and supplied by Calgon Corporation—a cationic polymer (W-2923) to break the emulsion and an anionic polymer (POL-Z-E 2706) to enhance coagulation. A single polymer system was recommended and supplied by Grace Dearborn (GD) Corporation—a cationic polymer (KLAR-AID 2400) as a coagulant. Two types of experiments were performed: jar tests and larger scale batch-mixing tank (BMT) experiments. O/G and TSS removal efficiencies were 99% for both the Calgon and GD polymer systems. The range of optimum dosage was broader for the Calgon polymers, but both systems were susceptible to overdosing. A longer settling time was required for the GD polymer, but the Calgon polymers were more sensitive to pH. Average residual production rates were 89 and 148 gallons per 1000 gallons of wastewater treated for Calgon and GD polymers, respectively.

INTRODUCTION

Coolant used in metal rolling operations is a complex mixture containing primarily fats and mineral oil, with lesser amounts of free fatty acids,

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emulsifiers, bactericides, and rust inhibitors. Emulsifiers are active components which reduce the interfacial tension of the surface of oil droplets. Wastewater used in this study was from a hot rolling mill and contained oil/grease (O/G) from coolant sprayed on metal strips to dissipate heat. The wastewater consisted of approximately 95% water and 5% emulsified oil. The O/G wastewater underwent phase separation in two oil recovery ponds. The pond effluent used in this study contained between 1080 and 3271 mg/L O/G and 200 to 1500 mg/L total suspended solids (TSS).

BACKGROUND

In an oil-in-water (O/W) emulsion system, surface-active agents accumulate at the oil–water interface and reduce interfacial tension to promote formation of an emulsified solution. The emulsified oil droplet is 2 to 3 μm in diameter and can be dispersed by uniform electronegative charges on the surface of each oil droplet. The term "stability" refers to the capacity of an oil droplet to remain an independent entity within the dispersion. The surface charge on the oil droplet produces a force of mutual electrostatic repulsion between adjacent droplets. If the charge is high enough, the oil droplets will remain discrete, dispersed, and in suspension. Reducing or eliminating the charge has the opposite effect—the droplets will agglomerate and rise to the wastewater surface. An emulsified waste can be destabilized by adding coagulants such as inorganic acids, inorganic salts, or synthetic polymers (1). The effectiveness of the chemical addition–gravity separation (CA–GS) process for an emulsified oil wastewater depends on: 1) the inherent stability of the system toward coagulation/flocculation, and 2) determining the appropriate chemical type/dosage and mixing and settling conditions (2).

The polymer method for O/G wastewater treatment is similar to the inorganic coagulant method and involves the addition of highly charged cations to destabilize negative surface charges for the colloid. Polymers with a longer hydrocarbon chain and straighter chain are preferred as coagulants (3). Depending on the nature of the particular polymer being used, polymers may adsorb ionically and through van der Waals forces. Charge density, ionic strength, molecular weight of a particular polymer, and extent of mixing influence the destabilization mechanism and resulting floc formation (4). The degree of destabilization depends on the type of polymer and wastewater treatment system applied. Charge density of the polymer fluctuates with water pH. When using a polymer(s), pH adjustments are occasionally required (5). The high ionic strength aids adsorption by reducing electrostatic repulsion if the polymer and the particles are of opposite charge. The higher molecular weight polymers, which

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signify long chains, function by interparticle bridging and can serve as an effective coagulant. However, polymers with molecular weight in excess of 10^7 are difficult to dissolve (6). Mixing is essential for a polymer to be adsorbed evenly on the surface of contaminated particles. Mixing at the time of polymer addition should be vigorous but of short duration because floc particles may be ruptured and become fragmented by prolonged agitation.

A dual polymer system may be used if a single polymer does not provide effective coagulation and flocculation. When two polymers are added to wastewater, flocculation is enhanced (7). Magdassi and Rodel reported that floc aggregate size and flocculation rates are larger when dual polymers are used (8). A cationic polymer plus an anionic polymer provides a means of tailoring floc size, floating characteristics, and shear strength. A cationic polymer is a charged neutralizing coagulant in a long chain, with a high molecular weight. It operates via mechanical bridging in addition to charge neutralization (9). An anionic polymer also has a high molecular weight and is often used for promoting growth of floc through mechanical bridging (10, 11).

Advantages of using polymers over inorganic coagulants include: 1) polymers produce larger and stronger floc, 2) a smaller volume of sludge is produced using polymers, 3) polymers are generally effective over a broad pH range, and 4) sludge produced with polymers tends to dewater more easily than sludge produced with inorganic coagulants. Disadvantages of the polymers include: 1) recovered oil/grease, although containing less water than with inorganic coagulants, may have a tight oil bond that requires costly subsequent oil de-emulsification; 2) polymer effectiveness can be more dependent on wastewater characteristics; and 3) because polymer chemistry is generally proprietary, wastewater treatment operators may be unable to easily troubleshoot system problems. Thus, either a well-trained operator or reliable, on-site assistance by the polymer supplier is required (12).

Jar testing is a relatively quick method for selecting the chemical type, dosages, and treatment conditions (i.e., mixing intensity and time, settling time). The major advantages of jar testing are that a small volume of material is required, testing time is short, and test replication is relatively inexpensive. A disadvantage of jar testing is the scaling errors that can occur. During full-scale operation, jar testing can be used to check chemical feed rates which may lead to a substantial decrease in the amount of chemical(s) used and sludge produced (13), as well as a better quality effluent. A well-planned jar-testing program is especially important when polymers are used because a polymer's effectiveness can be highly dependent on the influent characteristics.

Following chemical addition, the destabilized colloidal material (sludge) is removed using solid–liquid separation technologies (e.g., settling, dissolved air flotation). In this study, gravity settling was investigated. The effectiveness of the gravity settling process is a function of the hydrodynamic conditions and the chemical and physical characteristics of the effluent from the CA process (e.g., floc size, strength, and density).

INVESTIGATIVE APPROACH

Experiment Design

Two polymers recommended by Calgon Corporation, a cationic polymer (W-2923) and an anionic coagulant aid (POL-E-Z 2706), were investigated. The Calgon cationic polymer [molecular weight (MW) 8500] was used to break the oil emulsion while the anionic polymer (MW 10×10^6) was used to enhance the coagulation. Based on recommendations from Grace Dearborn (GD) representatives, a single cationic polymer, KLAR-AID 2400 (MW about 5×10^5) was investigated. Characteristics of the chemicals, as supplied by the manufacturer, are presented in Table 1. Before each experiment, stock chemical solutions were diluted to the appropriate concentration for easy chemical delivery and good distribution in the wastewater.

The effectiveness of the CA–GS process was investigated using jar tests (1000 mL) and batch-mixing tank (BMT) (1500 L) experiments. Jar tests were used to determine the optimum chemical(s) dosage for subsequent BMT tests. The importance of pH and influent characteristics were also evaluated. The BMT tests were used to evaluate O/G removal and the effectiveness the CA–GS process on a larger scale (relative to 1000 mL jar tests).

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TABLE 1
Characteristics of Polymers Used in the Experiments

Company	Trade name	Product classification	Density (g/mL)	pH	Water solubility
Calgon	W-2923	Cationic coagulant	1.22	2.4–3.2	High solubility
	POL-E-Z 2706	Anionic coagulant aid	1.00	5.7[a]	Miscible
Grace Dearborn	KLAR-AID 2400	Cationic coagulant	1.18	2.3–3.0	Appreciable

[a] 1% solution.

Materials and Methods

Jar Tests

Before starting a jar test, wastewater from the oil recovery ponds was transferred to two cleaned storage tanks and a sample was taken for pH, temperature, O/G, and TSS analyses. Five or six 1-L beakers were then filled with 1000 mL of the pond effluent. For Calgon chemicals, a cationic polymer was added into the beakers in 1 mL increments, resulting in a 100 mg/L incremental dosage between beakers. Typical ranges for the cationic polymer concentration after delivery were between 500 and 1000 mg/L. After cationic polymer chemical addition, the solution was mixed at 100 rpm for 2 minutes and then allowed to settle for 5 minutes. After settling, the turbidity of the supernate and the residual volume were measured. A predetermined amount of anionic polymer was then added to each jar. The anionic polymer dosage ranged from 5 to 20 mg/L. The solution was mixed at 100 rpm for 2 minutes and allowed to settle for 5 minutes, and the supernatant turbidity and the residual volume were measured. For several jar tests, the Calgon polymers were added simultaneously to determine if the use of a single full-scale chemical injection system was feasible.

Jar test procedures used during the GD polymer testing were similar to those used for Calgon polymers. Typical dosage ranges for the GD polymer were between 300 and 800 mg/L. Initially, a 2-minute mixing (100 rpm) and a 5-minute settling time were employed. However, with these times the performance of the GD polymer was marginal thus, the mixing time was increased to 6 minutes and the settling time was increased to 10 minutes.

The effect of pH on chemical dosage was also investigated by collecting 1000 mL samples of wastewater in which the pH was adjusted (pH range 5.4 to 8.6) using either NaOH or HNO_3. A predetermined amount of chemical(s) was added and the jar test was conducted as described previously.

Batch-Mixing Tank (BMT) Experiments

BMT experiments were conducted immediately after the completion of a series of jar tests. For the Calgon polymers, the BMT was filled with 1500 L of wastewater from the storage tanks, the appropriate amount of cationic polymer (determined by jar-testing) was added to the tank in the slug mode, and the contents of the tank were mixed for 20 minutes and then settled for 20 minutes. Following settling, the volume of sludge and supernatant turbidity were measured. A predetermined amount of anionic

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polymer was then added in the slug mode, and the BMT contents were mixed for 20 minutes and settled for 20 minutes. In addition to measuring residual volume and supernatant turbidity, the O/G, TSS, and pH of the supernate were determined. An identical procedure was used for the GD polymer except that there was no anionic polymer addition.

For several BMT tests the effect of settling time on turbidity removal and floc formation was investigated. Samples were taken from BMT immediately after polymers addition/mixing and allowed to settle for 5, 10, 15, and 20 minutes. After each settling time the residual volume and supernatant turbidity were measured.

Analytical Methods and Quality Assurance/Quality Control

O/G concentration was determined using Standard Methods 413.1, Liquid Partition Gravimetric method. TSS concentration was determined by Standard Methods 160.2. Turbidity and pH were determined using a Nephlometric Turbidimeter and an Omega pH meter, respectively. The upper detection limit for turbidity was 200 NTU. Temperatures were determined with a mercury thermometer. Ten percent of the analyses were duplicated. For each set of duplicated samples, deviations of each measurement from the mean of the two measurements as a percent of the mean were generally less than 10%.

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RESULTS AND DISCUSSION

Jar Tests

Chemical Dosages

In Figs. 1 and 2, results from a typical jar test are presented for Calgon and GD polymers, respectively. For the Calgon dual polymer system jar test, the final turbidity was 4.3 NTU and the optimal chemical dosages were 800 mg/L for cationic polymer and 10 mg/L for anionic polymer. The cationic polymer is a charged neutralizing coagulant which operated via mechanical bridging in addition to charge neutralization with the negative charged emulsified oil droplets. However, the floc formed was in a fine and loose suspension state. After anionic polymer addition, both the turbidity and residual volume were lowered significantly (data not presented). The long chain, high molecular weight anionic polymer adsorbed the fine floc and promoted bridge formation between floc, and fine oily floc was aggregated into a larger size which rose to the liquid surface. For the GD polymer jar test, the final turbidity was 22 NTU and the optimal chemical dosage was 600 mg/L. The GD cationic polymer MW was greater than that of the Calgon cationic polymer. It not only neutralized

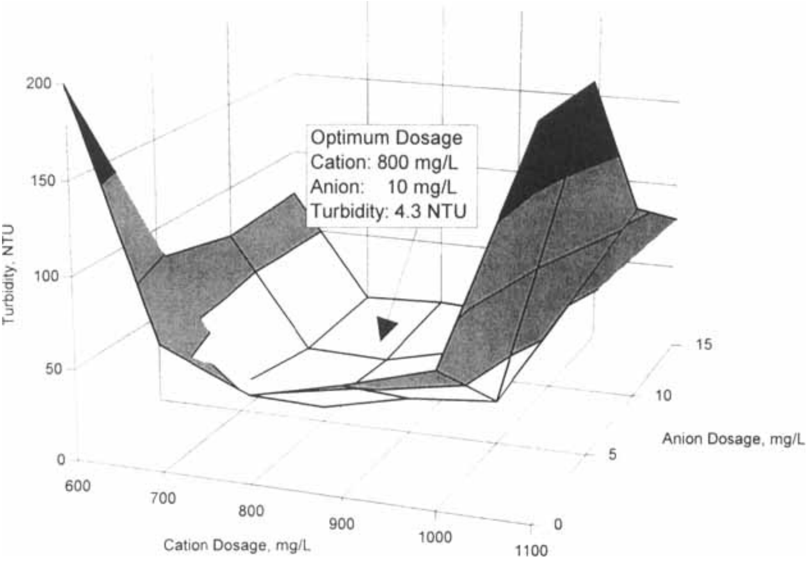

FIG. 1 Typical jar test for Calgon polymers.

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oil droplet surface negative charge but also promoted bridging formation between the floc. The dosage range where optimal turbidity removal occurred was small, and poor removal occurred for both chemical systems if the polymers were under- or overdosed. Therefore, for a full-scale CA-

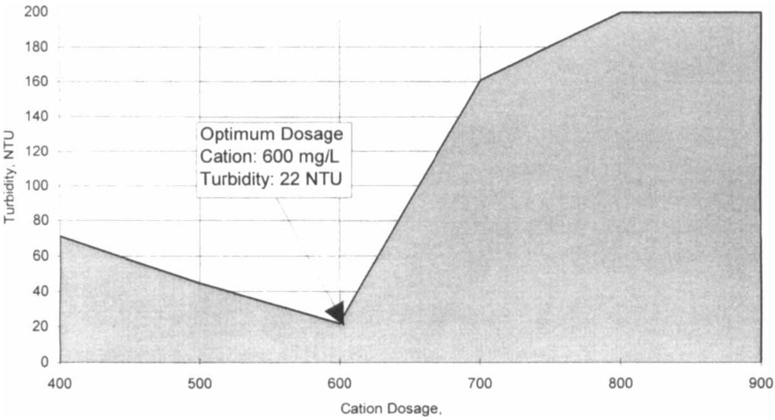

FIG. 2 Typical jar test for Grace Dearborn polymer.

GS system, frequent jar testing would be required. In Table 2, a summary of jar test results using Calgon and GD polymers is presented. Under a wide range of pond effluent conditions, the optimal dosages for the Calgon chemicals ranged from 500 to 1000 mg/L for the cationic polymer and from 5 to 25 mg/L for the anionic polymer. The optimal dosage for the GD chemical ranged from 300 to 700 mg/L. The final turbidity for the Calgon dual polymer system was, on average, about 4 NTU lower than that for the GD single polymer system.

In Fig. 3 the dosage occurrence percentages for the Calgon and GD chemicals are presented. For the Calgon dual polymer system, a cationic polymer dosage of 800 mg/L and an anionic polymer dosage of 5 mg/L had the highest occurrences at 30 and 38%, respectively. A cationic polymer dosage of 500 mg/L for GD chemical had the highest occurrence at 49% followed by 400 mg/L at 34%. The Calgon dual polymer system had a broader ranger of optimum dosages compared with the GD chemical. Given the inherent variability of the wastewater, the use of the Calgon dual polymer system in a full-scale system would be more difficult because of the increased possibility of under- or overdosing the system. For several jar tests the Calgon polymers were added simultaneously to determine if the use of a single full-scale chemical injection system is feasible. Generally, the emulsion was not broken, and therefore separate injection/mixing systems would be required.

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Relationship between Optimum Chemical Dosage and Influent Characteristics

In order to determine if the optimum dosage was related to wastewater characteristics such as pH, TSS, and O/G, a linear regression analysis

TABLE 2
Summary of Jar Test Results

Item	Calgon	Grace Dearborn
Dosage, mg/L:		
Cation	500–1000	300–700
Anion	5–25	NA[a]
Turbidity, NTU:		
Low	1	7
High	30	27
Mean ± 1 SD	11 ± 7	15 ± 6

[a] NA: Not applicable.

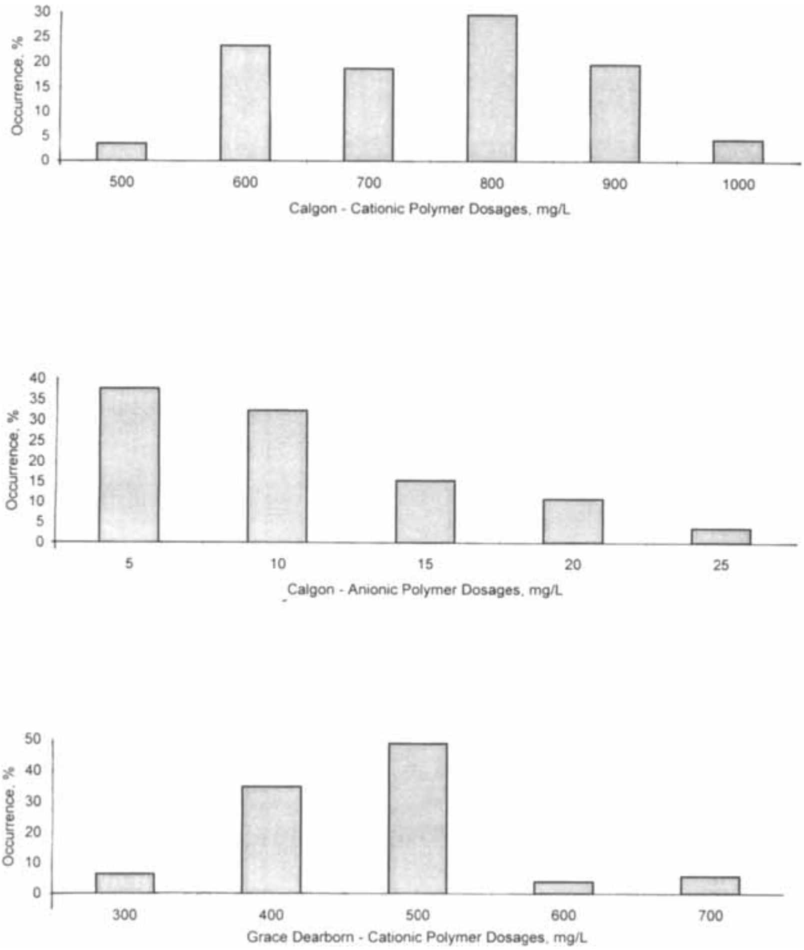

FIG. 3 The dosage occurrence percentages for the Calgon and Grace Dearborn polymers.

was performed. Results from the correlation analyses are presented in Table 3. The coefficient of determination, r^2, is the proportion of variation in the dependent variable explained by the linear relationship (14). $r^2_{0.95,v}$ is the coefficient of determination at 95% confidence interval under v degrees of freedom. Since all r^2 values in Table 3 are smaller than $r^2_{0.95,v}$, there were no statistically significant relationships between optimum chemical dosage and influent wastewater characteristics. Thus, appropriate chemical dosages cannot be estimated using influent pH, O/G, and

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TABLE 3
Results from Regression Analyses between Chemical Dosage and Influent Wastewater Characteristics

Dependent variable	Independent variable	Degree of freedom, ν	r^2	$r^2_{0.95,\nu}$
Calgon:				
Cationic polymer dosage	O/G	18	0.01	0.20
	TSS	18	0.00	0.20
	pH	25	0.01	0.15
Anionic polymer dosage	O/G	18	0.03	0.20
	TSS	18	0.01	0.20
	pH	25	0.00	0.15
Grace Dearborn:				
Cationic polymer dosage	O/G	11	0.06	0.31
	TSS	11	0.00	0.31
	pH	11	0.01	0.31

TSS concentrations, and jar tests are essential in determining the optimum dosage during full-scale operations.

Effect of pH Adjustment

In Fig. 4, supernatant turbidity versus pH for the typical Calgon chemical dosage–pH-dependent jar test is presented. The pH of the six jars was adjusted to between 6.4 to 8.6. The cationic polymer dose was 700 mg/L and the anionic polymer dose was either 5 or 10 mg/L. Supernatant turbidity was highly dependent on pH. The optimum pH was about 6.7, and turbidity was high at pH values greater and less than this value. There was little difference in turbidity at 5 and 10 mg/L of anionic polymer. In Fig. 5, supernatant turbidity versus pH for a typical GD chemical dosage–pH-dependent jar test is presented. The pH ranged from 5.4 to 8.4, and a 500 mg/L chemical dose was used. Below a pH of 7, turbidity removal was not dependent on pH. However, above pH 7 turbidity increased dramatically. Regardless of which chemical(s) is ultimately chosen, a pH control system should be included in the design of the full-scale process.

Batch-Mixing Tank (BMT) Experiments

There were 27 BMT experiments (15 for the Calgon dual polymer system, 12 for the GD single polymer system). The chemical dosage used during BMT experiments was determined from jar testing. A summary of

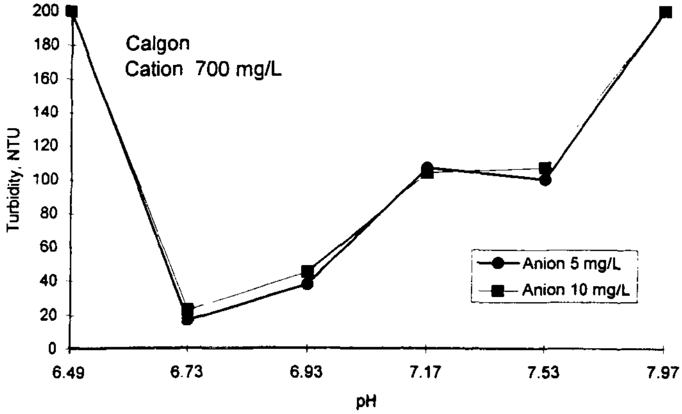

FIG. 4 Supernatant turbidity versus pH for a typical Calgon dual polymer–pH-dependent jar test.

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results from BMT experiments using Calgon and GD polymers are presented in Table 4. Influent O/G and TSS during BMT testing using Calgon polymers averaged 2255 ± 826 and 970 ± 333 mg/L, respectively and those for the GD polymer averaged 2400 ± 735 and 790 ± 404 mg/L, respectively. Significant amounts of O/G and TSS were removed by both the Calgon and GD polymer systems—average removal was 99% for both

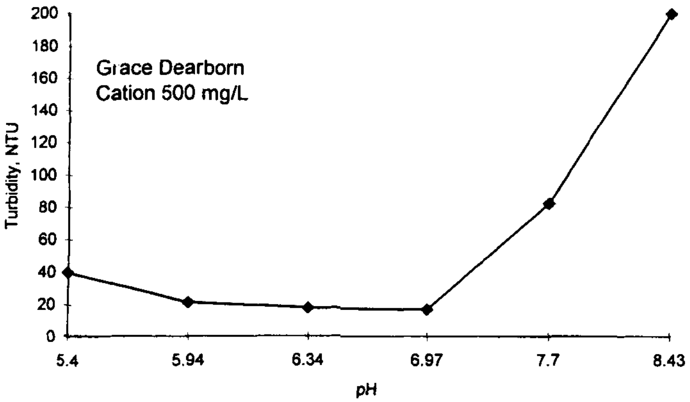

FIG. 5 Supernatant turbidity versus pH for a typical Grace Dearborn polymer—pH-dependent jar test.

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TABLE 4
Summary of Results from Batch-Mixing Tank Experiments

Parameter	Calgon	Grace Dearborn
Influent:		
O/G, mg/L	1080–3270	1080–3120
	(2255 ± 826)[a]	(2400 ± 735)
TSS, mg/L	200–1500	200–1430
	(970 ± 333)	(790 ± 404)
Effluent:		
Turbidity, NTU:	2–21	2–31
	(11 ± 6)	(11 ± 9)
O/G, mg/L:	3–52	1–35
	(15 ± 14)	(10 ± 5)
TSS, mg/L:	5–30	5–96
	(11 ± 7)	(20 ± 27)
Chemical dosages:		
Cationic polymer, mg/L	500–1000	300–700
Anionic polymer, mg/L	5–25	NA[b]
Residual production gal/1000 gal wastewater	50–130	70–120
	(89 ± 26)	(148 ± 42)

[a] Mean ± 1 SD.
[b] NA: Not applicable.

O/G and TSS. However, the discharge permit for the facility in question is envisioned to have an O/G limit of about 25 to 30 mg/L. Neither chemical system was able to consistently meet this limit. Thus the use of a tertiary process in a full-scale operation will likely be required to keep the facility in compliance. Supernatant turbidities after addition of both Calgon polymers ranged from 2 to 21 with an average of 11 ± 6. The supernatant turbidity prior to anionic addition ranged from 13 to greater than 200 NTU (data not presented). The anionic polymer promoted bridge formation between floc, and fine floc was aggregated into a larger size which rose to the liquid surface. Addition of the anionic polymer promoted bridging formation between the particles. For the GD chemical, turbidity ranged from 2 to 31 NTU (11 ± 9). As in the jar tests, turbidities were slightly lower when using the Calgon chemicals.

The volume of residuals generated by Calgon polymers ranged from 50 to 130 gallons (89 ± 26) per 1000 gallons wastewater treated and from 70 to 200 gallons (148 ± 42) per 1000 gallons for the GD polymer. The volume of sludge generated by the GD polymer was about twice that generated

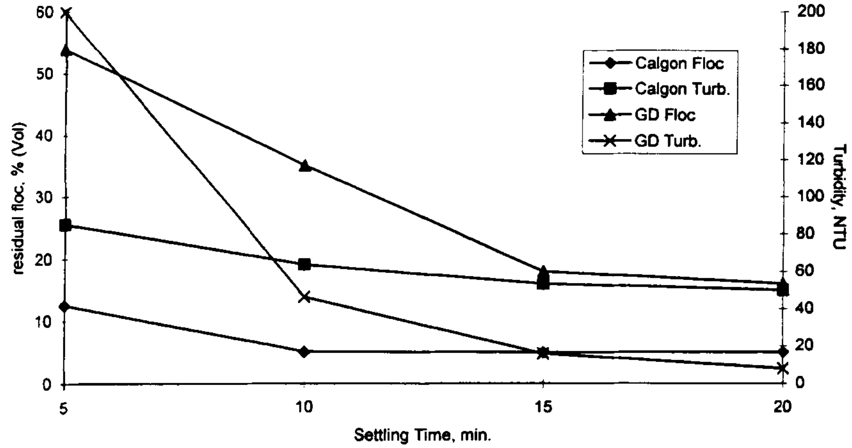

FIG. 6 Residual volume and supernatant turbidity versus settling time.

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by Calgon polymers. Despite GD personnel recommendations, the use of an anionic polymer to enhance flocculation by promoting bridging between floc particles may be desirable for reducing residual production.

In Fig. 6, residual volume and supernatant turbidity versus settling time are presented for two typical BMT tests. For Calgon polymers the residual volume decreased slightly with time. Residual volumes ranged from 10 to 15% of the total wastewater volume after 5 minutes of settling and leveled off at about 5% with settling times greater than 5 minutes. Turbidity decreased slightly with time. Thus, for the Calgon polymer system, settling time was not a critical parameter. In contrast, the effect of settling time on the performance of the single GD polymer system was dramatic. Supernatant turbidity after 5 minutes of settling was always above the detection limit of 200 NTU. After an additional 5 minutes of settling, turbidity decreased to less than 200 NTU, and after 20 minutes of settling the turbidity levels were similar to those observed during jar testing. Residual volumes ranged from 25 to 100% (i.e., emulsion was not broken) after 5 minutes of settling and decreased to between 12 to 20% after 20 minutes of settling. Thus, the settling time is a critical parameter for the GD polymer and the use of an anionic polymer to promote flocculation may be warranted.

SUMMARY

Wastewater from hot rolling mill operations that underwent phase separation was treated by organic polymer addition and gravity separation.

Recommended polymers from two corporations were investigated. Calgon Corporation supplied a cationic polymer (W-2923) to break the emulsion and an anionic polymer (POL-Z-E 2706) to enhance coagulation. Grace Dearborn Corporation supplied a cationic polymer (KLAR-AID 2400) as a coagulant. Although the characteristics of the polymers provided by the two corporations are proprietary, the MW of the GD polymer is greater than that of the Calgon cationic polymer and less than that of the Calgon anionic polymer. This provides some insight regarding the relative O/G and TSS removal performance and residual production. Both companies' chemicals were effective in reducing (99%) the O/G and TSS concentration of the wastewater. The effluent O/G concentration ranged from 3 to 52 mg/L for the dual Calgon polymer system and from 1 to 35 mg/L for the single GD polymer system. O/G reduction is especially important because the facility's future discharge permit will focus on this parameter. The optimum chemical dosages for Calgon polymers ranged from 500 to 1000 mg/L (cationic polymer) and 5 to 25 mg/L (anionic polymer), and for the GD polymer ranged from 300 to 700 mg/L. Based on a statistical analysis, the optimal chemical dosage was not related to influent wastewater characteristics (e.g., O/G, TSS, and pH). Optimum removal occurred in a relatively narrow dosage range, and the possibility of under- or overdosing was high. Thus, jar testing is essential for determining or adjusting the chemical dosage in a full-scale system. The volume of residuals produced using the GD single polymer system was about twice that observed when using the Calgon dual polymer system. A longer settling time was required for the GD chemical. Both systems were sensitive to pH, with the Calgon system being more sensitive. Regardless of which system is ultimately chosen, however, a pH control should be included in the design of the full-scale process.

Neither chemical system tested in this study consistently met the effluent discharge permit requirement. Even though the GD single polymer system resulted in a lower effluent O/G concentration, the Calgon dual polymer system resulted in less residual. While the effluent O/G concentration is most important for the facility discharge permit, the amount of residual to be disposed of can be an important cost consideration. Thus, any facility considering these alternative treatment polymer systems will have to examine both the effluent quality and the residual quantity, as well as the cost of any tertiary treatment required for compliance.

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