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Revision Memo

12/01 Addition of specific ion removal technology and other changes for clarification.

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SCOPE

This section presents background, design considerations, and guidelines for preparing duty specifications for facilities to separate free oil and suspended matter from wastewater by chemical flocculation and clarification. These units are typically located downstream of the primary oil/solids removal equipment and upstream of biological oxidation (BIOX). This section also addresses dissolved ion removal (i.e. metals, phosphate) by Adsorption/Coprecipitation, which is another chemical flocculation process using clarification equipment. Process, design, and operating considerations are given for the clarifier and for auxiliary facilities, including those for chemical addition.

ExxonMobil Engineering has not fixed many of the design parameters for these units; those pertaining to the clarifiers that have been fixed are given in **PREPARING THE DUTY SPECIFICATION** and in **DG 11-4-2**. The material preceding that is mainly descriptive in nature, and is intended to provide background.

Some information on the handling and addition of chemicals to assist wastewater clarification is included in this section, additional information can be found in the Design Practices and Design Guides listed below.

Sludge handling, thickening, drying, and disposal are also important considerations, and while not covered in this section, must also be included in the engineering. Gravity Sludge Thickeners are covered in [Section XX-A2](#).

Although clarifiers are also used for clarification of biological solids and raw water, no consideration is given in this section to these applications; and the information contained in this section should not be applied indiscriminately to these other clarification applications. Refer to [Section XIX-A7](#).

Jar tests should be run and a pilot test considered before a chemical flocculation unit is specified or designed for an existing refinery or chemical plant. For most grass roots designs, samples will not be available and units should be specified based on existing similar services, with provisions for increased operating flexibility.

REFERENCES

DESIGN PRACTICES

- [Section XIX-A](#) Guidelines for Selecting Wastewater Treatment Systems
- [Section XIX-A2](#) Flotation Units
- [Section XIX-A5](#) Biological Treatment of Wastewater
- [Section XIX-A7](#) Clarification Systems for Biological Treatment and Wastewater
- [Section XIX-A9](#) Water/Wastewater Chemical Feed Systems
- [Section XX-A2](#) Gravity Sludge Thickeners
- [Section XXVI-A](#) Boiler Feedwater Treating

EMRE WATER AND WASTEWATER DESIGN GUIDE (TME 080)

- DG 11-4-2 Cold Process Water Treaters
- DG 11-6-1 Chemical Feeders for Boilers and Deaerators
- DG 11-6-3 Chemical Feeders for Wastewater Treating

OTHER REFERENCES

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14. Havran, C. M., *Process and Design Considerations for Solids Contact Reactor/Clarifiers from Westech Engineering*, 2001 ECS2 R 500.
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16. Weber Jr, W., *Physiochemical Processes for Water Quality Control*, pp. 61 - 110 (1972)

DEFINITIONS

Alkalinity

The capacity to react with acids. Alkalinity is different from pH. Alkalinity is normally determined by titration with acid using methyl orange as an indicator, and is often reported as "M.O. or m alkalinity."

Coagulant

A chemical added to the water for the purpose of causing fine particles to agglomerate.

Coprecipitation

Removal of a contaminant by incorporation as an impurity within the matrix of precipitates composed primarily of other substances, physically entrapped within their pore spaces, or adsorbed on their surfaces.

Flocculant

A substance that causes coagulated particles to agglomerate into settleable flocs.

Launder

A water trough. Launder is sometimes used to denote the trough provided in a clarifier to collect the supernatant liquid.

Rise rate

One measure of the throughput of a settler, obtained by dividing the volumetric flow rate by the surface area of the basin.

Sludge

The precipitate and impurities that settle to the bottom of the clarifier or thickener.

BACKGROUND

In this section chemical flocculation or chemfloc means a process to remove suspended impurities (both oil and solid) or dissolved ions from water by the addition of chemicals to the water to form a floc, to adsorb dissolved ions (if applicable), and the subsequent settlement by gravity of the floc and attached impurities.

PROCESS SEQUENCE

Chemical flocculation consists of the following four sequential steps:

Coagulation - The provision of suitable chemicals (or coagulants) and high energy mixing to neutralize charges, destabilize particulates, and begin the formation of flocculent particles (or floc). This is sometimes termed **flash-mixing**, as flash mixing is typically used to achieve coagulation. In some cases the added chemicals are already in the form of a floc; in other cases they form a precipitate.

Flocculation - The provision of retention time with gentle agitation to allow the floc particles to precipitate, to associate with the impurities, and to increase in size by interparticle bridging, entrapment, and continued aggregation. This step is sometimes called **slow-mix**.

Sedimentation - The gravity settling of the floc.

Removal of the Settled Material - After coagulation, flocculation, and sedimentation, the floc is removed as a sediment or sludge.

Although chemical flocculation can be carried out batch wise, it is usually a continuous flow process.

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BACKGROUND (Cont)

COAGULANT ACTION

The action of the coagulant is complex. The following four different mechanisms may be involved:

Mechanical Entrapment - Many coagulants are acidic materials (salts of aluminum, iron, etc.) and react with the alkaline components in the water to form hydrate precipitates; sometimes a hydrate precipitate ($\text{Mg}(\text{OH})_2$) is added directly. These precipitates carry down the suspended solids by mechanical entrapment. The precipitates also act as nuclei for the start of further precipitation. The solid particles and also much of the oil are trapped by the enormous surface area of the floc. Some of the oil, however, usually floats free in the settling step.

Electric Charge Neutralization - Counter-ions may exert a charge-neutralizing effect on the electrically charged colloidal particles, thus allowing the particles to agglomerate and settle.

Reaction - A coagulant material, such as lime, may be added that reacts chemically with dissolved components in the water yielding a settleable solid.

Adsorption - The precipitates formed by coagulants have a tremendous surface area, which can effectively adsorb colloids and some dissolved ionic species.

In this section, the suspended floc and the absorbed material are jointly referred to as a single solid for simplicity.

USES AND EFFECTS OF CHEMICAL FLOCCULATION

Uses - Chemical flocculation can be used to remove suspended matter, free oil, and some types of dissolved materials from water. The coagulation agent accelerates the settling of suspended material and often allows removal of solids not separated by conventional sedimentation. Chemical flocculation, for example, may allow removal of contaminants which, because of their small size or unfavorable specific gravities, fail to settle unaided or settle so slowly that gravity separation alone is impractical. Suspended, colloidal, non-settleable, and certain dissolved matter can be removed from water by chemical flocculation. Chemical flocculation is not normally used on streams having settleable amounts of free oil. An example of using chemical flocculation to remove dissolved matter, is the adsorption or coprecipitation of dissolved metals by iron hydroxide floc. This process is called Iron Coprecipitation.

When chemical flocculation is used to remove oil and suspended solids, it is normally carried out downstream of primary oil and solids removal, e.g. API separator or corrugated plate interceptor. In general, consider using a chemfloc unit to separate oil and suspended solids from wastewater if the suspended solids in the influent are heavier than water but settle too slowly to be separated by simple settling.

- When used to remove specific ions, chemfloc units may be located at the source of a specific wastewater stream or after the biological treatment unit (BIOX). Obviously if the chemfloc unit is on a specific wastewater stream it will be smaller than if it is treating the entire BIOX effluent, however issues such as interference from oil and the oxidative state of the ion must be considered. Examples of specific ion removal are: coagulation with iron salts, lime or alum to remove phosphate, coagulation with iron salts to remove metal ions, coagulation with iron salts to remove sulfide.

The chemical flocculation units that have been installed for wastewater treating in ExxonMobil affiliated refineries serve:

- To protect a downstream biological oxidation unit from excessive influxes of oil.
- To protect downstream carbon adsorption columns from excessive oil and solids.
- To increase the cycle time of downstream deep-bed sand or mixed-media filter.
- To reduce the oil content of API separator effluent prior to final discharge.
- To remove stabilized, fine oil droplets that are ineffectively removed by gravity separation.
- To reduce color and turbidity.
- To remove some COD and BOD associated with the non-dissolved material.¹
- To reduce the concentration of sulfides (by using iron salts).
- To remove selenium (in the form of selenite) from wastewater prior to discharge (Iron coprecipitation).

Effects - The chemfloc operation should produce an outlet water essentially free of entrained oil and suspended and settleable solids. The effluent water can be expected to contain less than 10 ppm turbidity (NTU).

¹ Dissolved organics do not respond appreciably to coagulation; so usually the BOD of the water is hardly affected by the chemical flocculation process.



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BACKGROUND (Cont)

CONTAMINANT REMOVAL EFFICIENCIES

Well-designed chemfloc units can have removal efficiencies as listed below:

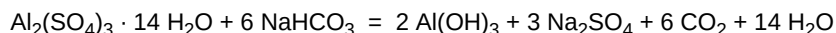
Free Oil	> 90%
Suspended Solids	> 90%
Hydrogen Sulfide	> 95%, dependent on pH
Specific Ions	up to 99%

♦ COAGULANTS AND COAGULANT AIDS

Polymeric Coagulants and Aids - Polyelectrolytes are the most commonly used coagulant/coagulant aids. There are literally thousands of polyelectrolytes available; including cationic, anionic, and neutral forms. Collectively, polyelectrolytes are usually referred to as "polymers". Cationic polymers are usually used as primary coagulants, while anionic and non-ionic polymers are used as coagulation aids. Jar tests determine the form most suitable for a particular waste and contaminant. The polymers are delivered as a concentrated solution which should be diluted with softened water or demineralized water if available. Typically, polymers do not function properly when diluted with treated water.

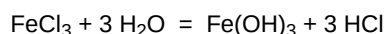
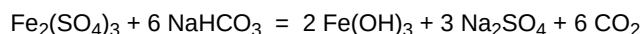
Polymers are discussed in detail in the *Polyelectrolyte Guide*, ER&E Report No. EE.20E.84 (1984), and **Section XIX-A9 - Water / Wastewater Chemical Feed Systems**.

Alum - Alum (Aluminum Sulfate) is probably the most commonly used inorganic coagulant. It may be used to remove both oil and solids, and specific ions, e.g. phosphate. It is available both as a solid and as an aqueous solution (see **DG 11-6-3**). The alum solution may require dilution; if so, it is diluted to the optimum concentration (determined by testing) with the water to be treated. Alum is used in the municipal water treating industry but it is not commonly used in the petroleum industry. Alum is corrosive, and piping and equipment in alum solution service should be fabricated of corrosion-resistant materials. In general, when alum is the coagulant, a pH of about 6.5 to 7.5 is required. Alum produces an aluminum hydroxide floc and consumes alkalinity when it dissociates, as shown in the following reaction with sodium bicarbonate:



For every 1 mg of alum, 0.26 mg of insoluble $\text{Al}(\text{OH})_3$ is produced and 0.51 mg of alkalinity (as CaCO_3) is consumed.

Iron Salts - Iron salts, usually ferric chloride, ferric sulfate or ferrous sulfate, are used as coagulants to remove sulfide or phosphate, or to coprecipitate metals from wastewater. The function of the iron is to form a high surface area iron hydroxide floc, which adsorbs suspended solids, oil, and certain forms of heavy metal ions from the aqueous solution. If sulfide or phosphate are present, the iron will complex with these ions, forming FeS and FePO_4 instead of $\text{Fe}(\text{OH})_3$. This is a removal process if sulfide or phosphate removal is desired, or a competing process if metal ion removal is desired. The capacity of the iron floc for metal ion removal is very dependent on pH, the form of the heavy metal, and the inlet and outlet concentrations of the metal to be removed. Use of iron salts for metals removal is discussed in more detail in the Iron Coprecipitation section. Iron floc is relatively "light" especially at low pH's; therefore, rise rates should be selected conservatively. Proposed rise rates greater than 0.6 gpm/ft² (0.012 m/hr) should be justified by settling tests or pilot clarifier tests. Free oil can inhibit settling of iron floc. The simplified chemical reactions producing the iron hydroxide floc and consuming alkalinity are as follows:



For every 1 mg of ferric sulfate, 0.53 mg of insoluble $\text{Fe}(\text{OH})_3$ is produced and 0.75 mg of alkalinity (as CaCO_3) is consumed. For every 1 mg of ferric chloride, 0.65 mg of insoluble $\text{Fe}(\text{OH})_3$ is produced and 0.93 mg of alkalinity (as CaCO_3) is consumed.

Magnesium Oxide - Magnesium oxide, which forms a magnesium hydroxide floc, can be used to remove silica by adsorption.

Polyaluminum Chloride - A flocculent more widely used in Japan for suspended solids, oil and grease and phosphorous removal, has the advantage that it does not reduce alkalinity to the extent of other inorganic coagulants.

Bentonite Clay² - Bentonite clay can be used in chemfloc units, although no ExxonMobil affiliates are known to do so. Using inorganic coagulants may have an impact on the ash balance of some disposal outlets. Bentonite clay is available both in pulverized form and in granular form. Continuous clay metering/slurrying equipment is available. The clay slurry concentration should be kept low; a 5% slurry of Thixo-Jel is quite viscous and is the maximum concentration that should be prepared.

² Clay of the sodium montmorillonite (90%) type (e.g., Georgia Kaolin Thixo-Jel #1).

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BACKGROUND (Cont)

Activated Silica - Activated silica is an effective coagulant aid. Unfortunately, it has an extremely short self-life; and when used, it is usually manufactured at the unit, and this is a separate process. If activated silica is used, addition facilities and also preparation facilities are usually required unlike when other coagulant aids are used. Activated silica is prepared from sodium silicate and sulfur dioxide. The sulfur dioxide neutralizes the alkali in sodium silicate-either wholly or partially.

Others - New developments in this area include proprietary organic polymers and inorganic complexes, such as silicates, for use in specific ion removal.

♦ IRON COPRECIPITATION

Selective ion removal of metals using iron salts is referred to as Iron Coprecipitation, even though the mechanism is primarily by adsorption rather than by precipitation. Iron Coprecipitation is effective at removing trace metals down to ppb levels, but iron dosage is in the ppm range. Therefore, sludge production and handling is definitely a consideration. The process involves addition of iron salts to the wastewater, which form ferric hydroxide precipitate.

To minimize clarifier size, improve kinetics and possibly minimize sludge production, the iron coprecipitation process should be installed on the wastewater source stream containing the ion to be removed. However, it may be necessary to install it downstream of biological treatment due to interference of organics, sulfide and phosphate with adsorption of the specific metal(s). Sulfide and phosphate react with the iron salts to form FeS and FePO₄ instead of ferric hydroxide. Also, the metals may need to be in the oxide form to be removed by iron coprecipitation.

Whether or not a metal species is removed and at what pH strongly depends on the oxidation state of the metal. Removal of cations increases with increasing pH (see **Figure 1**). Removal of anions increases with decreasing pH. Increasing the iron dosage will shift the curves somewhat, i.e. allow removal at lower pH for cations and higher pH for anions. Obviously from the curves, if several metals need to be reduced, the optimum pH and iron dosage must be identified. It may not be possible to adequately remove 2 or more metals at the same conditions, for example simultaneous removal of selenium and nickel.

Dose-response jar testing is strongly recommended to determine the iron dosage and pH due to potential interferences in the wastewater. A minimum of about 10 - 15 mg/l of iron may be needed to produce sufficient floc. **Figure 2** shows the results of dose response jar testing for selenium removal from Benicia wastewater. Most of the selenium was in the form of selenite, or Se(IV) oxidation state. Jar testing is also used to determine the polymer (coagulant aid) type and dosage and to give an indication of coagulation, flocculation, and settling properties.

Metal ion removal is primarily by adsorption on the iron hydroxide floc. This is somewhat reversible if the pH changes, so solids must be separated from the wastewater at the optimum pH. A coagulant aid, generally a high molecular weight anionic polymer, is needed to enhance floc formation and settling, because the iron hydroxide floc is light.

Addition of ferric chloride lowers the pH of the water, so pH may have to be adjusted to achieve the desired metal removal. For example, to removal copper or nickel would require the pH to be increased. In the case of selenite removal, the pH may not have to be further adjusted because selenite removal works best at slightly acidic pH.

♦ SOLIDS PRODUCTION IN IRON COPRECIPITATION

In order to size sludge handling facilities and estimate disposal costs, the amount of solids that will be produced must be known. This can be **estimated** by calculation, but should be confirmed by jar testing, and this will include the indigenous wastewater solids and the solids produced by the chemical addition. The solids produced by chemical addition include the primary coagulant, the coagulant aid, the metal removed (often negligible because it is at the ppb level), and any additional precipitates due to the pH change. For example, if the pH is being increased, calcium carbonate or magnesium hydroxide may be precipitated. Once the solids production is known, the amount of sludge can be calculated. The sludge stream from the clarifier may contain only 1% - 2% solids. Solids concentration in the sludge can be more precisely estimated from jar or pilot testing. To calculate the solids produced, use the following equation:



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BACKGROUND (Cont)

$$M = M_I + M_C + M_P + M_A \quad \text{Eq. (1)}$$

$$M = Q \cdot (C_I + C_C + C_P + C_A) \cdot 0.012 \quad (\text{Customary})$$

$$M = Q \cdot (C_I + C_C + C_P + C_A) \cdot 0.024 \quad (\text{Metric})$$

where:

- M = Total mass of solids produced in the iron coprecipitation process, lb/day (kg/day)
- M_I = Mass of solids coming in with the influent wastewater, lb/day (kg/day)
- M_C = Mass of solids produced by the primary coagulant, lb/day (kg/day)
- M_P = Mass of additional precipitate formed due to pH adjustment, lb/day (kg/day)
- M_A = Mass of solids produced by the coagulant aid, lb/day (kg/day)
- Q = Feed Wastewater flow rate, gpm, (m³/h)
- C_I = Concentration of solids in the influent wastewater, wppm (mg/L)
- C_C = Concentration of the precipitate produced by the primary coagulant, wppm (mg/L)
- C_P = Concentration additional precipitate produced, wppm (mg/L)
- C_A = Concentration of the coagulant aid, wppm (mg/L)

Below is an example using ferric chloride for selenium removal. The total mass of solids produced by removal of 70 wppb selenite from 1500 gpm of wastewater containing 30 wppm suspended solids using ferric chloride at 40 wppm as iron, with 4 wppm polymer addition is calculated as follows:

$$M(\text{lb/day}) = 1500 \text{ gpm} \cdot (30 \text{ wppm} + \left(40 \text{ wppm Fe} \cdot \frac{1.91 \text{ wppm Fe(OH)}_3}{\text{wppm Fe}} \right) + 4 \text{ wppm}) \cdot 0.012$$

$$M = 1987 \text{ lb/day}$$

Note that in this example, no additional precipitate (M_P) was produced.

EQUIPMENT TYPES AND DETAILS

DESCRIPTION OF THE UNIT

A chemical flocculation unit consists of five major parts (**Figure 3**):

- Chemical storage tanks, metering pumps, and injection points
- Flash-mixing (contacting of coagulants with contaminants)
- Flocculation
- Clarification
- Sludge handling, treatment, and disposal

The flash-mixing, flocculation, and clarification can be carried out in separate vessels, but are most often accomplished in separate compartments of a single vessel (**Figures 4 and 5**).

♦ SOLIDS CONTACTING

Solids contacting is usually employed to enhance flocculation. Solids contacting allows some of the previously formed precipitate to act as "seed" or nuclei for new precipitates. This results in particles that are fewer, larger, and more easily settled than would otherwise be the case.

Solids contacting can be accomplished in two ways: by the use of a sludge blanket (**Figure 4**) and by recirculation (**Figure 5**). In sludge blanket clarifiers, upward flow in the settling zone is used to suspend a deep bed of sludge (sludge blanket) and provide contact of the influent water and chemicals with solids. This is accomplished by allowing the effluent from the flocculation chamber to flow through the sludge blanket in the main clarification chamber. It is important that the sludge blanket be kept at the proper level for good results. These units do not tolerate rapid flow variations and best results are obtained in the 60% - 90% capacity range.