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Jar Test for Precipitants, Coagulant and Flocculants

Precipitation is the chemical conversion of soluble substances (including metals) into insoluble particles. Coagulation and flocculation causes a chemical reaction that promotes the formation, agglomeration or clumping of such particles to facilitate their removal from solution.

The amount or dosage of a precipitant, coagulant and/or flocculant required to precipitate and remove metals in wastewater solutions is not only dependent on the concentration of such metals in solution, but also on several other factors. To optimize the dosage, the following parameters must be considered:

- The solution pH.
- The chemical used to adjust the pH (i.e. NaOH, lime, $Mg(OH)_2$, Na_2CO_3).
- The different types (and concentrations) of metals present in solution.
- The amount and types of chelants and complexing agents present in solution.
- The amount of residual oxidizers present in solution.
- The coagulants and flocculants used.
- The sequence in which chemicals are added.

JAR TEST PROCEDURE

As indicated above, untreated process wastewaters may contain ingredients other than dissolved metals that will affect the treatment methodology. Therefore, the procedure which follows provides a starting point and adjustments may be required to achieve the desired results.

RECOMMENDED EQUIPMENT:

1. pH meter with electrode to monitor pH.
2. ORP meter with electrode to monitor the reduction reaction.
3. 300 ml - 400 ml Beakers, clear plastic or glass.
4. Magnetic Stirrer or equivalent.
5. Eyedroppers for adding chemical reagents.
6. Laboratory Type Filter.
7. Metals Test Kit or AA Spectrophotometer, etc.

CHEMICAL REAGENTS:

1. Sodium-Hydroxide (Caustic-Soda) solution.
2. Sulfuric-Acid solution.
3. Precipitant(s).
4. Coagulant solution(s) – see product bulletin for suggested dilution ratio.
5. Flocculant solution(s) – see product bulletin for suggested dilution ratio.

PROCEDURE:

1. Pour a sample of untreated wastewater into a beaker (ex 300 ml). While mixing, adjust the pH using caustic soda or sulfuric acid to the optimum pH for hydroxide precipitation of mixed metals, i.e. pH 8.5.
2. While stirring the sample, use an eyedropper to add the precipitant until the ORP value drops rapidly by 150mV (typically to -250 mV). If an ORP meter with electrode is not available, use several beakers and add different amounts of precipitant to each beaker.

Please note that, although the precipitating reactions appear to be instantaneous, a retention time of up to 15 minutes may be required to obtain a complete reaction.

3. Add 1 or 2 drops of coagulant solution. Mix at high speed for 1 to 3 minutes. Turn off mixer and observe the coagulation (agglomeration) of the precipitated particles. If the particles appear to be coagulating but need assistance to accelerate their settling, a flocculant may be added to the solution followed by a slow mixing to allow for floc building. If the settling action is too slow or incomplete, redo the test and add a drop or 2 of an iron or aluminum coagulant before the initial pH adjustment in #1 above.

4. After several minutes a sample of clear supernate may be taken for metals test, or the entire contents of beaker(s) may be filtered to remove solids, then the filtrate tested/analyzed.

Note: When the supernate has a yellow or orange tint (similar to the color of the precipitant), that is an indication of overdosing the precipitant. If necessary, dilute the precipitant before adding it to the wastewater. Make note of the dilution ratio for determining the optimum full-scale dosage. Overdosing the precipitant can also cause a significant increase of colloidal particles and interfere with the normal coagulation/flocculation reactions.

To project the dosage results from a jar test to full scale, the following information may be helpful:

1 drop = 0.05 ml

1 drop per liter = 50 mg/l (ppm)

PRECIPITANT CONTROL SYSTEM

Ecologix Environmental Systems can recommend control systems for dosing precipitants. Such systems utilize an electrode for accurate measurement (in millivolts) of the reduction reaction of precipitants in wastewater. The electrode signal is monitored by the controller that controls the metering pump for dosing the required amount of precipitant. Systems are also available for pH adjustment under electrode control.

pH

The solubility of metallic particles is pH dependent. That is, dissolved heavy metal ions can be precipitated chemically by adjusting the pH of a wastewater stream. The pH is important because all metals have a pH at which their solubility is minimal. Although this pH differs for all metals, it generally lies between 7.5 and 11. Since most wastewaters contain a variety of metals, it is difficult choosing the optimum pH at which their solubility is minimal.

Ecologix precipitants will simultaneously precipitate a variety of metals at any given pH within the above range. These precipitants, with low solubility, can achieve very high removal efficiencies.

When used as a "polishing" precipitant, the dosage of a precipitant can be lowered depending on the quantity of metals that are precipitated as hydroxides by pH adjustment. While a pH of 8.5 is normally recommended for the polishing effect -- the pH value will vary depending on the presence of chelating and/or complexing agents in the wastewater. A jar test procedure as described above can help to establish the optimum pH within the 7.5 to 11 range.

For adjusting pH, sodium hydroxide is recommended. However, other common chemicals can be used -- such as soda ash and lime. For some applications (nickel complexes) magnesium hydroxide is an effective reagent.

RESIDUAL OXIDIZERS

When a precipitant is added to a wastewater stream containing residual oxidizers along with dissolved metals, dual reactions occur. These reactions are: 1) the reduction of the oxidizers and 2) the precipitation of the metals. This, of course, increases the amount of precipitant required for total metals precipitation.

To optimize the usage of a precipitant, adding a reducer (i.e. sodium metabisulfite) before the precipitant addition will remove the oxidizers from the wastewater solution.

FLOCCULANTS/COAGULANTS

Characteristically the insoluble particles formed by adding a precipitating reagent to a wastewater solution are very small and suspended in the solution (colloidal). The suspended stability of such particles is due to both their small size and to the electrical charge (usually negative) on their surface causing them to repel their neighboring particles.

To promote the removal of these suspended solids requires chemical coagulation and/or flocculation. Adding coagulants to the wastewater creates a chemical reaction in which the repulsive electrical charges surrounding colloidal particles are neutralized, allowing the particles to stick together creating clumps or flocs. The aggregation of these particles into larger flocs permits their separation from solution by sedimentation, flotation, filtration or straining. When required, flocculants with an anionic charge are commonly used to facilitate the agglomeration of the flocs and their settling.

Some precipitants contain cationic polymers that neutralize the precipitated particles. The cations (positive charges) from the polymer reduce or reverse the negative charges of the precipitate which, in turn, permits the coagulation and flocculation of the particles.

COMPLEXING AND CHELATING AGENTS

Some process wastewaters include complexing and chelating agents, which bond to the metal ions making precipitation difficult, if not impossible, for many precipitating reagents.

Ecologix precipitants are capable of breaking many of these bonding agents and thereby precipitating the metal ions without the addition of other chemicals. In some instances a combination of pH adjustments and varying reaction times may be required along with a precipitant and flocculants.

ODOR CONTROL

By oxidation, some precipitants have a characteristic sulfide odor. This odor may be eliminated or minimized by following simple control procedures:

- a. Assure that the waste stream or sludge contains no oxidizers.
- b. Dose the precipitant below the wastewater surface to prevent surface air oxidation.
- c. Use below surface mechanical agitation for mixing. Do not use air agitation.
- d. Do not overdose. The Reagent Control System mentioned above will prevent overdosing.
- e. Maintain a wastewater pH of 7.0 or above.
- f. Utilize mechanical exhaust ventilation.
- g. Use closed top reaction/mixing tanks when possible.
- h. After wastewater clarification, any sulfide odor or color is a positive sign of overdose. If impractical to control dosing, excess may be easily removed with a minimal injection of peroxide or hypochlorite in the effluent.

Please note that many Ecologix precipitants include an odor neutralizer that neutralizes sulfide and other odors.