Coagulation and flocculation

Jartest
Framework
This module explains the lab experiment on coagulation and flocculation.

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1. Objective

The objective of this experiment is to achieve a quality improvement of surface water by using an iron salt as coagulant. Turbidity is the quality parameter to be improved. When dosing an iron salt a pH of 8 is expected to be optimal for reducing the turbidity.

2. Experiment set-up

A jar test apparatus, used for this experiment, is schematically drawn in figure 1. It consists of six identical beakers of 2 liter, each equipped with a stirrer, two dosing test tubes and a siphon with Erlenmeyer flask (not drawn). One dosing test tube is for acid or alkaline dosing, the other for dosing the coagulant. They can be operated simultaneously. The stirrers are driven by one motor, thus giving each the same rotation velocity.

Adding ferric chloride causes a number of hydrolysis reactions, where the iron ions react as an acid, thereby reducing the pH. As a result of these reactions iron hydroxide already precipitates in an acidic environment. In a $2 \times 10^{-4}$ M solution (about 11 mg Fe/l) the formation of insoluble iron hydroxide starts at pH=2.7, while at pH=4 even 99.85% of all available iron will be precipitated.

The hydrolysis products interact with colloidal matter and finely dispersed particles. They are destabilized, thereby causing coagulation and flocculation. The application of coagulants can also be used for reducing the color or the phosphate concentration.

In order to optimize the flocculation process a number of process parameters are relevant:
- coagulant dose
- pH
- shear rate during flocculation
- flocculation time
- dosing of a coagulant aid (a polymer)

The shear rate can be calculated with:

$$G = \sqrt{\frac{P}{\mu \cdot V}}$$

$P$ = mixing energy, dissipated power (W)
$\mu$ = dynamic viscosity (N·s/m²)
$V$ = volume of mixing tank (m³)

3. Theory

Colloidal matter in surface water can not settle by itself. Dosing of a coagulant is required to achieve removal by coagulation and subsequent flocculation, followed by sedimentation. The result of this treatment is depending on many factors, such as coagulant dose, pH, reaction period and supplied energy of the stirring device. A jar test is ideal to assess the influence of these process parameters within a short time.
4. Procedure

4.1 Variation of iron dose at a fixed pH=8
In order to maintain pH=8 in each beaker after the dosing of different quantities of iron (FeCl$_3$.6H$_2$O), it is necessary to add some NaOH to compensate the effect of the iron salt on pH. The amount of NaOH depends on the iron salt dosage and must be estimated before the jar test is executed.

- fill a beaker with 1.8 L Schiewater, put it on a magnetic stirrer and immerse the pH-electrode; stir continuously.
- measure the pH and add with a burette 0.5M NaOH until the pH=8.
- note the added quantity of NaOH (ml).
- add with a burette five times 2 ml FeCl$_3$-solution and adjust after each iron addition to pH=8 by adding NaOH. Note the added quantities.
- calculate the cumulative amount of NaOH that is needed at iron doses of 0, 2, 4, 6, 8 and 10 mg Fe/l.

4.2 Variation of FeCl$_3$ dose at final pH=8 at 30 rpm
- measure and note the temperature of the Schiewater and adjust the thermostat on this temperature to effectuate a constant temperature during the test.
- fill each beaker with 1.8 L Schiewater.
- fill six test tubes with 0, 2, 4, 6, 8, and 10 ml FeCl$_3$-solution and fix them in the clamps above the beakers. The zero dosing is the blank determination.
- fill six other test tubes with NaOH as determined in the previous paragraph and fix the tubes in the other clamps.
- add simultaneously the NaOH to the beakers, while stirring intensively at 200 rpm; rinse the test tubes with some demineralised water.
- add simultaneously, at the same stirring intensity of 200 rpm, the coagulant and rinse the test tubes with some demineralised water. Stir for 10 to 15 seconds.
- reduce the stirring intensity to 30 rpm and stir gently for a period of 20 minutes. Examine the floculation phenomena that occur in the beakers. Note the appearance of the first visible flocs, floc shape and size.
- after 20 minutes of slow stirring turn off the stirrer and let the solution stand for 20 minutes to enable settling of flocs.
- take carefully with a siphon a sample of about 500 ml from each beaker and collect it in a flask of 1000 ml.
- determine of each sample the turbidity and pH.

4.3 Variation of FeCl$_3$ dose at final pH=8 at 60 rpm
- measure and note the temperature of the Schiewater and adjust the thermostat on this temperature to effectuate a constant temperature during the test.
- fill each beaker with 1.8 L Schiewater.
- fill six test tubes with 0, 2, 4, 6, 8, and 10 ml FeCl$_3$-solution and fix them in the clamps above the beakers. The zero dosing is the blank determination.
- fill six other test tubes with NaOH as determined in the previous paragraph and fix the tubes in the other clamps.
- add simultaneously the NaOH to the beakers, while stirring intensively at 200 rpm; rinse the test tubes with some demineralised water.
- add simultaneously, at the same stirring intensity of 200 rpm, the coagulant and rinse the test tubes with some demineralised water. Stir for 10 to 15 seconds.
- reduce the stirring intensity to 60 rpm and stir gently for a period of 20 minutes. Examine the floculation phenomena that occur in the beakers. Note the appearance of the first visible flocs, floc shape and size.
- after 20 minutes of slow stirring turn off the stirrer and let the solution stand for 20 minutes to enable settling of flocs.
- take carefully with a siphon a sample of about 500 ml from each beaker and collect it in a flask of 1000 ml.
- determine of each sample the turbidity and pH.
5. **Elaboration**

Execute the following steps:

- present the results in tables.
- what water quality parameter has an influence on the buffer capacity of water? Can you calculate the amount of that buffer in the Schiewater? You can either calculate it with the formulas in the lecture notes (softening) or use Stimela.
- explain why the extra NaOH-dose is necessary after dosing ironchloride. Is the extra amount in the range of what you expect?
- Plot in a graph the iron dose against the turbidity and residual iron concentration for the two experiments.
- give your comment on all results.
- calculate the optimal stirring rate at a water temperature of 2 degrees.
- at low temperatures the coagulation is often less efficient also at an optimal shear rate. What process parameters can be adapted in order to enhance the coagulation/sedimentation process at low temperatures?
Data form

Group number: Date:

Variable dosing FeCl₃ at constant end pH = 8
Temperature Schiewater: °C
Iron analysis extinction blank: 1/m

<table>
<thead>
<tr>
<th>Beaker</th>
<th>FeCl₃ sol. 1,8 g Fe/l ml</th>
<th>NaOH 0,5 M ml</th>
<th>Extinction Fe-meting 1/m</th>
<th>End quality water</th>
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<td>(pH) Turbidity (NTU) Fe (mg/l)</td>
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Constant dosing at variable end pH
Iron dosing (constant): mg Fe/l
Temperature Schiewater: °C
Iron analysis extinction blank: 1/m

<table>
<thead>
<tr>
<th>Beaker</th>
<th>FeCl₃ sol. 1,8 g Fe/l ml</th>
<th>Required end-pH</th>
<th>Dosage</th>
<th>Extinction Fe-meting 1/m</th>
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