Vincent Louvel 04/09/2024

Sodium Acrylate 38% (w/w) – Preparation Protocol

Purpose

To prepare ~180 mL of 38% (w/w) sodium acrylate solution by neutralizing acrylic acid with sodium hydroxide under controlled temperature.

Safety

WARNING: This procedure uses **concentrated acrylic acid** (corrosive, lachrymator) and **10 N NaOH** (highly caustic). Both can cause severe burns and permanent eye damage within seconds.

- Perform **all steps in a certified fume hood**.
- Wear **lab coat, safety goggles/face shield, and appropriate chemical-resistant gloves**.
- Use a **spill kit** and have **eyewash and safety shower** accessible.
- Neutralize and dispose of waste according to institutional regulations.

Reagents

- Acrylic Acid (Sigma-Aldrich, Cat. No. 8.00181)
- 10 N NaOH
- Deionized or double-distilled water (ddH_2O)

Equipment & Materials

- Glass beaker (250 mL or larger)
- Glass bottle (≥100 mL) for NaOH
- Magnetic stir plate and stir bar
- Peristaltic pump with compatible tubing
- pH meter with temperature probe
- Ice bucket / thin plastic container for ice bath
- Centrifuge (for later clarification), capable of 1000 × g
- Storage bottles (screw-cap, chemical-resistant)

Target Preparation

- Final volume: **≈180 mL**
- Composition (approximate):
  - 50 mL acrylic acid
  - 75 mL 10 N NaOH
  - 58 mL ddH_2O

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Procedure

1. Initial Mixing

1. In a clean glass beaker, add **58 mL ddH_2O**.
2. **Slowly** add **50 mL acrylic acid** to the water **while stirring**.
    - **Important:** Always add *acid to water*, not water to acid.
3. Add a magnetic stir bar and stir for ~1 min to obtain a homogeneous solution.

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2. Ice Bath & pH Probe Setup *(see setup picture)*

1. Place ice into a thin plastic container.
2. Insert the beaker (water + acrylic acid) into the ice, then add water around it to form an ice–water bath.
    - The beaker should be **approximately half immersed** in the ice–water mixture.
    - Add more ice as needed to keep the bath cold and stable.
3. Place the beaker on a magnetic stirrer and **start stirring**.
4. Install the pH/temperature probe in the solution, ensuring:
    - The probe is **fully submerged** in the liquid.
    - It does **not interfere** with the stir bar.

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3. NaOH & Peristaltic Pump Setup *(see setup picture)*

1. In a separate glass bottle, **pour 75 mL of 10 N NaOH**.
2. Connect the peristaltic pump tubing:
    - Place the **inlet/collector end** at the **bottom** of the NaOH bottle.
    - Secure the **outlet end** above the beaker in the ice bath.
3. Position the outlet so that it drips **into the beaker** but:
    - The tube **does not touch** the liquid.
    - There is enough clearance for the volume to increase during addition.

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4. Controlled Neutralization

1. Start the peristaltic pump at **~50% of its maximum rate** to begin NaOH addition.
    - The solution temperature will start to rise.
2. When the solution temperature reaches **25 °C**, **stop the pump**.
3. Restart the pump at **~10%** and closely monitor **both temperature and pH**.
4. Adjust the pump speed (typically **around 14%**) so that:
    - The temperature stays **near 25 °C** (avoid >30 °C).
    - The NaOH is added **gradually**, preventing overheating and overshooting the pH.
5. As the pH approaches **7.0**, **slow the pump further** and then:
    - **Stop the pump immediately** once the pH is slightly above neutral (pH 7.0–7.5).

If pH Overshoots (e.g., pH ≈ 10.5)

1. Remove the beaker from the ice bath and place it on the stir plate at **room temperature**.
2. Allow the temperature to stabilize while stirring.
3. Carefully add **small aliquots of acrylic acid** (e.g., 0.5–1 mL at a time) while monitoring pH, until it drops to **pH 7–8**.
4. Allow the solution to mix thoroughly after each addition before re-measuring pH.

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5. Equilibration, Clarification & Storage

1. Transfer the neutralized solution to a **closed container** (loosely capped if still slightly warm) and allow it to stand **overnight at room temperature** or 4 °C to equilibrate.
2. The next day, inspect the solution:
    - If **no precipitate** is visible, proceed to storage.
    - If **precipitate is present**, centrifuge at **1000 × g for 10 min** and collect the **clear supernatant**.
3. Aliquot the clear sodium acrylate solution into storage bottles.
4. **Storage recommendations:**
    - **Preferred:** −20 °C for long-term storage.
    - **Alternative:** 4 °C for shorter term use.
5. Label each container clearly with:
    - “Sodium acrylate 38%”
    - Date of preparation
    - Preparer’s initials
    - Storage conditions and any relevant hazard warnings.

Notes & Troubleshooting

- **pH target:** A final pH of **7.0–8.0** is typically suitable for downstream applications; adjust if your protocol requires a specific value.
- **Foaming:** If foaming occurs, reduce stirring speed while maintaining good mixing.
- **Ice bath:** If temperature creeps up, add more ice and/or reduce the pump speed.

Sodium Acrylate preparation
Guichard/Hamel Lab

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Picture of the set up: