

**Table:** Various etching and staining techniques on polished surfaces of clinker, slag, and cement (adapted from St. John et al. and Campbell)

| Outline of the Staining or Etching Method and Exposure Time  | Observation   |
|--|---|
| 10% Potassium hydroxide (KOH)  | Alite - Does not etch; Belite-Does not etch; C <sub>3</sub> A - Bluish gray; Ferrite-Bright white   |
| KOH + 5% Ethyl Alcohol, immersed for 20 secs, followed by washing in 1:1 ethyl alcohol plus water solution and then in isopropyl alcohol solution, buff for 15 sec on MicroCloth wetted with isopropyl alcohol | C <sub>3</sub> A turns blue   |
| NaOH (2.5 g) + ethyl alcohol (10 mL) + water (40 mL) solution, immersed for 10 sec, followed by washing in 1:1 ethyl alcohol plus water solution and then in isopropyl alcohol solution, no buffing            | Darkens alkali sulfates   |
| 10% boiling NaOH 20 sec immersion, wash with alcohol   | CA in high alumina cement turns blue or brown   |
| KOH (0.1 molar aqueous) immersed for 30 sec, followed by rinse with isopropyl alcohol spray and dry with forced warm air   | C <sub>3</sub> A and alkali aluminate turn blue-brown, alkali-sulfate darkens, free lime turns brown  |
| Dilute salicylic acid stain (0.2 g salicylic acid + 25 mL ethyl alcohol + 25 mL water), immersed for 20-30 sec., followed by alcohol spray wash  | Alite and belite are blue green; a solution with isopropyl alcohol (instead of ethyl alcohol) will provide a faster stain on alite than belite and hence distinguishes two phases   |
| Salicylic acid etchant (0.5 g salicylic acid in 50 mL methyl alcohol) 45 sec. etch.  | Alite and belite are clear, alite more strongly etched than belite, belite shows lamellar structure   |
| Nital (1.5 mL nitric acid in 100 mL ethyl, methyl, isopropyl, or amyl alcohol) etched for 6-10 sec.  | Alite and belite react quickly; alite turns blue to green, belite turns brown to blue – both show detail internal structure. Nital superimposed on a 20-sec KOH etch turns C <sub>3</sub> A light brown and colors the silicates. |
| HNO <sub>3</sub> in alcohol immersed for 2-15 sec.   | Alite - Grayish brown; Belite-Blue; C <sub>3</sub> A - Light gray; Ferrite-Bright white   |
| Hydrofluoric acid (HF) vapor (kept at 20-22°C) exposure for 5-10 sec.  | Alite - Buff brown; Belite-Blue; C <sub>3</sub> A - Light gray; Ferrite-Bright white; C <sub>2</sub> AS (melilite) in a high alumina cement is colored in HF vapor  |
| 10% Isopropyl Alcohol (10mL isopropyl alcohol plus 90 mL water), immersed for 30 sec.  | Reacts strongly with alite and weakly with belite; C <sub>3</sub> A shows a weak reaction   |
| Ammonium chloride (saturated, aqueous), 10 sec.  | Hexagonal section of alite perpendicular to the three-fold axis stains light yellow; prismatic section parallel to the c-axis turns blue; zoned alite shows light-blue core and dark-blue rim                                     |
| Ammonium chloride stain = 1g NH <sub>4</sub> Cl + 20 mL H <sub>2</sub> O + 20 mL ethyl alcohol + 10 mL acetone + 150 mL isopropyl alcohol, 10-20 sec.  | Alite turns brown, belite is unaffected. After 30-45 sec., Alite turns yellow to yellowish green, belite to brown   |
| Ammonium nitrate solution (1g NH <sub>4</sub> NO <sub>3</sub> + 20 mL H <sub>2</sub> O + 20 mL ethyl alcohol + 10 mL acetone + 150 mL isopropyl alcohol), 20-30 sec. etch.                                     | Alite turns yellow-green; belite turns brown  |
| Ammonium nitrate solution after the salicylic acid stain, 30 sec.  | Shows internal structure of alite and belite.   |
| Distilled water, 3-5 sec.  | Free lime (CaO) shows rapid etching from dark to iridescent green and blue. C <sub>3</sub> A turns dark blue. Alite becomes brown, belite is distinguished morphologically. Alkali sulfates are dark.                             |
| Water pH 6.8-7 on a saturated microlap cloth, 3 secs. Isopropyl alcohol wash, warm air dry   | MgO hard high physical relief, CaO turns dark to green/blue, C <sub>3</sub> A turns dark blue, C <sub>3</sub> S turns brown, C <sub>2</sub> S shows lamellar structure  |
| 10% MgSO <sub>4</sub> solution, etch for 60 secs at 50C  | C <sub>2</sub> S in slag shows striation.   |
| 1% borax solution, etch for 30 sec., wash with alcohol   | C <sub>12</sub> A <sub>7</sub> in high alumina cement turns gray  |
| Borax solution (1%, boiling) immersed for 10 sec.  | Etches blade-like pleochroite crystals in high alumina cement   |

**Table:** Various staining techniques in concrete, aggregate, and clinker raw feeds

| Specimen   | Method  | Observation   | Reference            |
|--|---|---|----------------------|
| Sulfates<br>Lapped, polished or thin sections  | Immerse in 2:1 mixture of BaCl <sub>2</sub> : KMnO <sub>4</sub> 6% solution for 2 minutes, wash first with water then with saturated oxalic acid.   | Ettringite, gypsum, anhydrite, plaster stain pink to purple.  | St. John et al.      |
| Feldspar, Quartz, and Calcite for staining raw feeds, aggregates, and thin sections  | Two minute exposure to concentrated HF vapor (52%), two minute immersion in sodium cobaltinitrite solution, washing in distilled water, 15 sec. exposure to 10 mL of barium chloride solution, washing in distilled water, two minute immersion in amaranth solution (28 grams of F.D. and C. Red No. 2 pure coal tar dye in 2 liters of water), quick washing is distilled water, and drying.  | Plagioclase feldspar (other than albite) stains red, potash feldspar stains yellow, quartz and glassy slags are unstained, calcite stains to various shades of pink, dolomite to deep red.  | Hutchinson; Campbell |
| Carbonates<br>Lapped surface<br><br>Thin sections  | 10 sec etching in 15% HCl;<br>30 sec immersion in 1:1 g Alizarin Red S + 0.9 g Potassium.<br><br>Ferricyanide in 100ml 1.5% HCl.<br>10 sec immersion in 0.2g Alizarin Red S in 100 ml 1.5% HCl, wash in water.  | Calcite stains pink<br>Ferroan calcite stains purple-blue.<br><br>Ferroan dolomite stains turquoise.<br>Dolomite is unaffected.   | Dickson in Hutchison |
| Evamy stain for calcite, ferroan dolomite, and ankerite applicable to aggregates, raw feeds and thin sections  | Immersion in 1% solution of HCL for 2-3 minutes (for thin section 0.2% solution for 30 sec), rinse with distilled water, flood with evamy solution and keep there for 2 minutes, wash with distilled water, dry with warm air. Evamy solution is prepared by mixing 1 gram of alizarin red S with 5 grams of potassium ferricyanide in distilled water, adding 2 ml of concentrated HCl and brining the final solution to 1 liter with distilled water, solution is stored in dark container. | Iron-free calcite stains red, iron-poor calcite stains mauve, iron-rich calcite stains purple, iron-free dolomite not stained, ferroan dolomite stained light blue, ankerite stains dark blue.  | Campbell, Hutchinson |
| Copper nitrate staining test for carbonate rock identification, classification (limestone versus dolomite) and detection of impurities including clay or shale inclusions or seams in limestone and dolomite | Immerse aggregates in 0.5M copper nitrate solution (prepared by dissolving 250 grams of copper nitrate trihydrate in 1 liter distilled water and filtering) for 16 hours; to convert stained calcite to the deep blue color, aggregates can be immersed in a strong ammonia solution for a few minutes. Finally aggregates are thoroughly washed in water and air dried.  | Splits in clay or shale seams; calcite or dense pure limestone stained deep blue, or greenish if limestone has iron impurities. Dolomite stains very light blue. Non-carbonate or silicate impurities are not colored. Argillaceous limestone or dolomite may show splitting or cracking. | Dolar-Mantuani       |

|  |  |   |                            |
|--|--|---|----------------------------|
| Methylene blue staining for detection of smectite in aggregates      | Prepare a methylene blue solution containing 4.5 grams of trihydrate methylene blue dye or 4.283 grams of dihydrate dye per liter of distilled water; a uncovered grain thin section containing different sieve fractions of aggregate is immersed in the dye solution in a petri dish for 5 minutes, rinse, and then dry. Another method is dye adsorption by finer than No. 200 sieve (75- $\mu$ m) fraction of sand grains in a titration process and spot testing of grains on a filter paper until a light blue ring forms around the central dark blue spot of the adsorbed grain. | Smectite particles turns blue, muscovite, biotite and other rock forming minerals do not stain, amount of blue-stained smectite grains can be determined by point counting. | Higgs                      |
| Alkali-silica gel  |  |   |                            |
| Fractured, grounded or thin section                                  | 15 min immersion in 10% uranyl acetate 1.5% acetic acid solution, wash in water.   | UV light at 240 nm wavelength gives yellow-green fluorescence of ASR gel.   | Natesaiyer and Hover       |
| Fractured, grounded or thin sections                                 | 72 hour absorption in 4M cuprammonium sulfate, wash in water.  | Gel exudations in voids and cracks stain blue.  | Poole, McLachlan and Ellis |
| Fractured, or aged surface   | Aqueous saturated solution of sodium cobaltinitrite followed by a counter stain with rhodamine B base solution.  | Bright yellow stain of alkali-rich ASR-gel; pink rhodamine stain of calcium-rich gel.   | Guthrie and Carey          |
| Highlighting microcracks by fluorescent dye mixed alcoholic solution | Treatment of a ground section of moist concrete with a fluorescent dye mixed alcoholic solution followed by rinsing excess solution, drying, and observation in a UV light.  | UV light at 240 nm wavelength gives yellow green fluorescence of microcracks.   | Gran, Mayfield             |

[Note: Consult the original reference before using a particular procedure for the first time]