

Inorganic Carbon Precipitation and Anaerobic Reactions in Agricultural Wastewater

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Summary

Water, sediment and gas phase sampling was conducted to evaluate the potential burial of inorganic carbon formed through sulfate and nitrate reduction in the Salton Sea and constructed wetlands in the Imperial Valley. Bicarbonate alkalinity, sulfide, and other chemical constituents were quantified in sediment pore water and the overlying water column, while gas phase concentrations of H₂S and NH₃ in the atmosphere were sampled with passive diffusion tubes. This analysis allowed for flux estimates to be made from the sediments to the overlying water column and from the water column to the atmosphere. In the Salton Sea, the calculated flux of H₂S+HS⁻ from the sediment pore water to the overlying water is 60,600 Mg y⁻¹; however, less than 1% appears to be volatilized. The production of HCO₃⁻ via sulfate and nitrate reduction, pyrite precipitation, and volatilization of H₂S and NH₃ increases the amount of calcite that is precipitating in the Sea by 70%, compared to the amount that would form if these reactions were not occurring. The total calcite precipitation in the Salton Sea is 578,000 Mg y⁻¹. This amount is 1.7 times the mass of calcite precipitating in the soils of the Imperial and Coachella valleys. Downstream reactions within a closed irrigation basin need to be considered when determining the changes in carbon storage due to irrigated agriculture.

Objective

The objective of this project is to quantify the rate of C storage as CaCO₃ due to anaerobic decomposition of organic matter via sulfate and nitrate reduction, and sulfide-mineral formation in the Salton Sea and constructed wetlands. These anaerobic processes produce “new bicarbonate” that drives calcite precipitation in flooded soils and lake sediments. These reactions are not generally considered when accounting for changes in carbon storage within an agricultural project.

Approach and Procedures

The Salton Sea water, sediment, and air were sampled at 12 locations along three transects of the lake from July 2005 to December 2006 (*fig. 1*). Sampling sites were located and repeatedly sampled using a Garmin eTrex global positioning system and the WGS84 datum. Water quality measurements were collected using a Hydrolab Sonde 4a calibrated following the manufacturer's instructions. Hydrolab casts provided dissolved oxygen (mg L⁻¹), temperature (°C), electrical conductance (mS cm⁻¹), oxidation-reduction potential (ORP in mV), and pH as a function of depth. Transparency was also measured in the field using a Secchi disk. Grab samples of the sediments were collected with a Ponar dredge and samples of the water column were collected (at the surface, at mid-depth, and just above the sediment) using a 2L Van Dorn sampler. Water samples were collected in triplicate at each depth. One set of water samples was collected in 125-ml bottles, which were completely filled with water, leaving no head space and immediately

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stored on ice. One set of water samples was acidified in the field with concentrated HNO_3 to $\text{pH} < 2$ and one set was preserved upon collection with 50% antioxidant buffer (APHA, 1998). All field samples were transported on ice to the laboratory until analysis.

Electrodes were used in the laboratory to monitor ORP and pH profiles of the sediment. Subsequently, the pore water was extracted from the sediments via centrifugation (12,000 rpm, 15 min.) and the resulting supernatant was filtered through a 0.45-micron polycarbonate membrane filter. The unfiltered pore water as well as the unfiltered overlying water column were each analyzed for alkalinity by titrating to an endpoint of pH 4.5. Concentrations of total S^{2-} were determined on the unfiltered pore water and the unfiltered overlying water column using an Orion IonPlus silver-sulfide ion-selective electrode on a Fisher Accumet mV meter as outlined in standard methods (APHA 1998). Following filtration through a 0.45 μm polycarbonate filter and acidification with 50% H_2SO_4 to $\text{pH} < 2$, the $\text{NH}_4\text{-N}$, $\text{NO}_3\text{-N}$, and $\text{PO}_4\text{-P}$ species were analyzed colorimetrically using a Technicon Autoanalyzer following standard methods (APHA, 1998). The major cations, including Ca^{2+} , Cu^{2+} , Mg^{2+} , Mn^{2+} , Na^+ , Si^{4+} , Sr^{2+} , K^+ and total Fe, were analyzed using an inductively coupled plasma spectrophotometer (Perkin-Elmer Optima 3000 ICP DV). Major anions, including SO_4^{2-} and Cl^- , were determined on the Dionex by ion chromatograph. Dissolved Fe^{2+} was determined using the colorimetric *o*-phenanthroline method on the Milton Roy Spectronic 601 spectrophotometer at a 510 nm wavelength.

Passive air diffusion sampling tubes were also deployed at various locations on the perimeter of the lake, which included the dock, the Salton Sea State Park, and a background location away from the lake (*fig. 1*). Sampling tubes were also deployed at 137 cm, 175 cm, 213 cm, and 251 cm above the surface of the water on a pier in the southern portion of the lake, identified as the Target in figure 1.

Sediment samples were extracted using several sequential extraction procedures to determine the concentrations of pyrite (FeS_2), amorphous FeS , greigite (Fe_3S_4), Fe(III) -oxides, and silicate Fe (deKoff et al. 2007).

CaCO_3 from sediments and barnacle shells growing in the Salton Sea were analyzed for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ isotope ratios to further verify the formation of HCO_3^- via anaerobic oxidation of organic matter.

Data from two constructed wetlands located on the banks of the New River in the Imperial Valley were analyzed to determine if sulfate reduction might be contributing to CaCO_3 precipitation. The Imperial wetlands located NE of Imperial off Wienert Rd are 43 acres (22 acres of wetted soil). The Imperial wetlands are sustained with agricultural drainage water from the Rice drain. The Brawley wetlands are 9 acres (6 acres of wetted soil) located SW of Brawley, and receives New River water. The inflows and outflows from the wetlands were sampled six times by the Imperial Irrigation District from 7/01 to 1/04 and analyzed for Cl , SO_4 , HCO_3 , Ca , Mg , Na , K , ClO_4 , NO_3 , NH_4 , PO_4 , total N, dissolved oxygen, pH, and suspended solids.

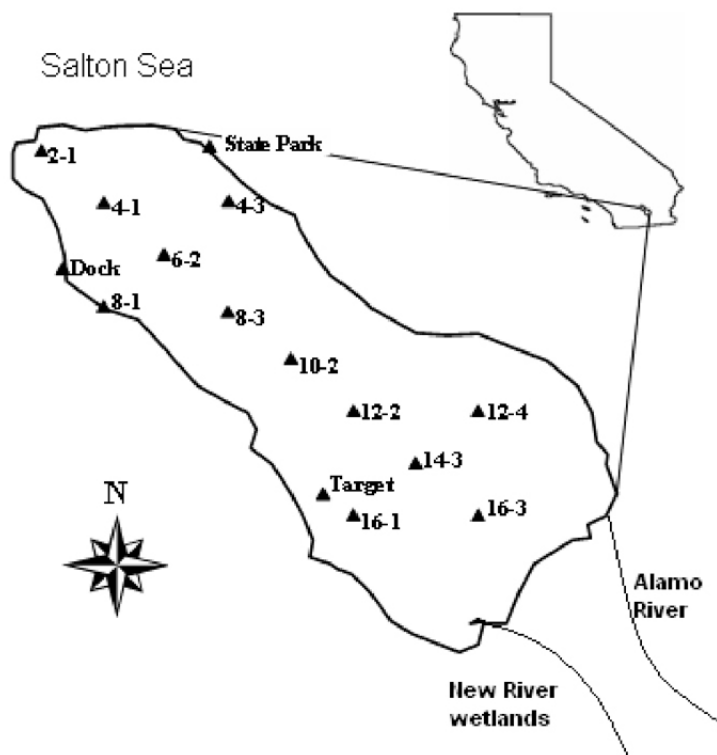


Figure 1. Passive air diffusion sampling tubes were deployed at various locations on the perimeter of the lake, which included the dock, the Salton Sea State Park, and a background location away from the lake.

Results

Salton Sea Water Column and Sediment Pore Water

Water column and pore water data collected at the Salton Sea were consistent with a eutrophic, hypersaline lake. Temperature profiles indicate that the Sea remained relatively well mixed from the fall through the early spring, after which rapid heating began from increased solar radiation (*fig. 2a*). Although the Salton Sea does not stably stratify for long periods of time, thermal stratification began in May 2006 when the difference in temperature of the surface to the bottom waters was more than 8°C (*fig. 3a*, ΔT). Stratification continued through the summer (approximately two months) until a strong wind event that began on 7/30/2006 mixed the lake, which resulted in zero dissolved oxygen throughout the water column (*fig. 2b*). The temperature gradient from the surface to the bottom during the well-mixed winter months was commonly less than 1°C (*fig. 3a*). The temperature profiles for the south basin and mid-lake locations showed similar seasonal heating and cooling patterns; however, the south basin appeared to have mixed more frequently as the bottom water at this site was found to be 1-3°C warmer on June 2 and August 11, indicating the warm surface water mixed to the bottom.

Anoxia in the bottom water (hypolimnion) was present throughout the summer, which contributes to the anaerobic processes such as sulfate reduction that generate alkalinity in the lake (*fig. 2b*). The ORP above the sediments was -150 to -250 mV, indicating strongly reducing conditions favorable for sulfate-reduction.

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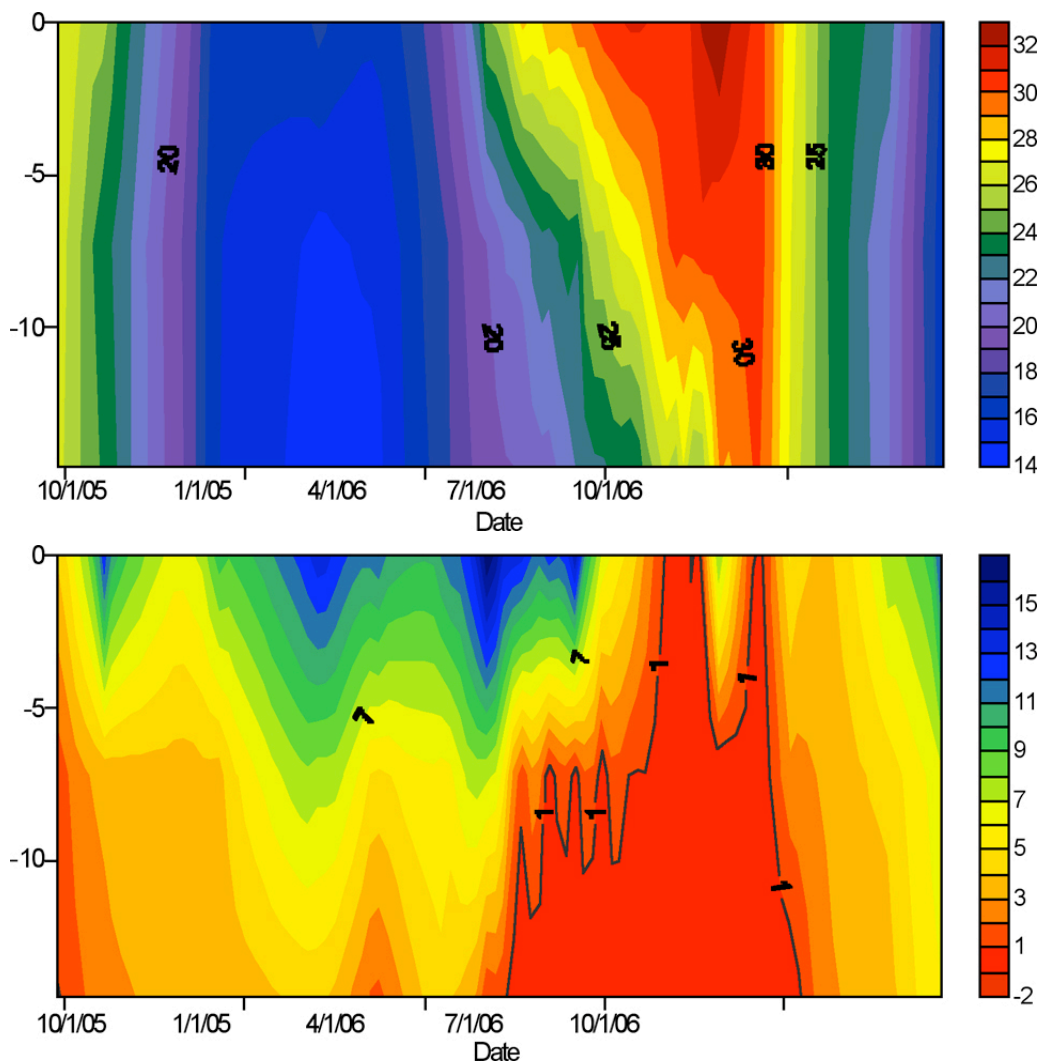
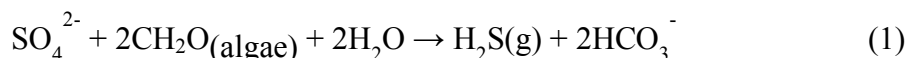


Figure 2. a) Water temperature ($^{\circ}\text{C}$) and b) dissolved oxygen (mg L^{-1}) as a function of depth and time of year for the north basin of the Salton Sea at site 6-2.

The sediment pore water had greatly elevated sulfide and alkalinity levels compared with the overly bottom water (*figs. 3 and 4*). Unlike the water column, however, the sediment of the Salton Sea remained anoxic and highly reduced throughout the entire year.

Sulfide concentrations always exceeded 1 mM in the pore water with concentrations of 5 mM measured in the warm summer months when the lake would thermally stratify (*fig. 3b*). Alkalinity levels were 2-4 times higher in the sediment pore water than the overlying water (*figs 3c and 4b*). These elevated bicarbonate concentrations are attributed to the anaerobic oxidation of algal biomass via sulfate reduction with the formation of H_2S (Eq. 1).



Additional evidence for bicarbonate production via sulfate reduction can be seen in the $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ isotope ratios for the sediment CaCO_3 and barnacle CaCO_3 . Barnacles growing in the surface waters of the Salton Sea had average $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ isotope values of -1.73 and +0.03 ‰, respectively, while the sediment CaCO_3 had δ -values of -2.06 and -4.37‰, indicating that some of the sediment CaCO_3 could be attributed to organic matter oxidation.

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The pH of the pore water was typically 0.8 to 1.0 pH unit lower than the overlying Sea water. These low pH values are attributed to the elevated CO₂ formed during the decomposition of the organic matter.

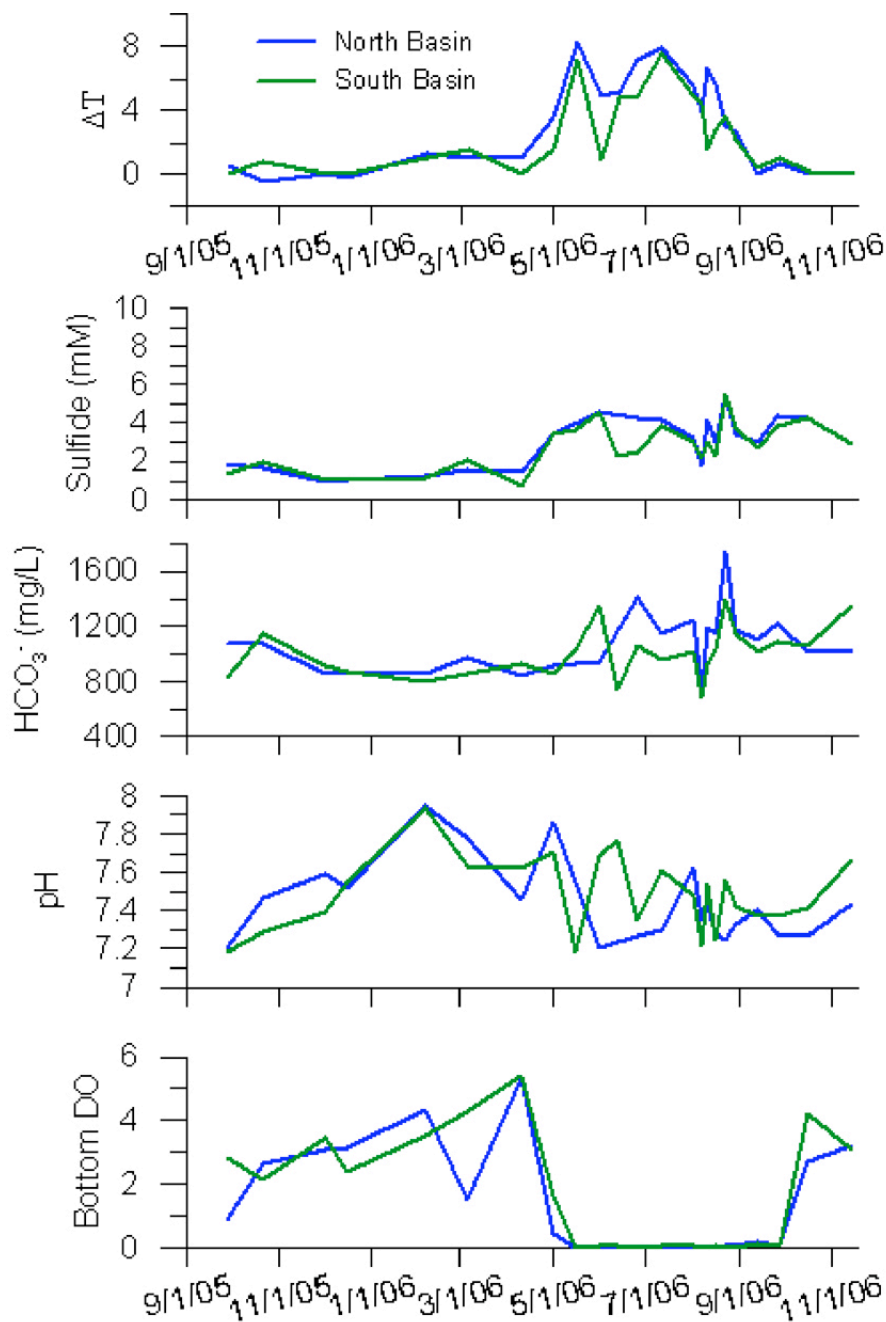


Figure 3. a) Temperature differential between the surface water and bottom water of the lake. b) Sediment pore water sulfide concentration and c) bicarbonate concentration and d) pH e) bottom water dissolved oxygen concentration in mg L⁻¹

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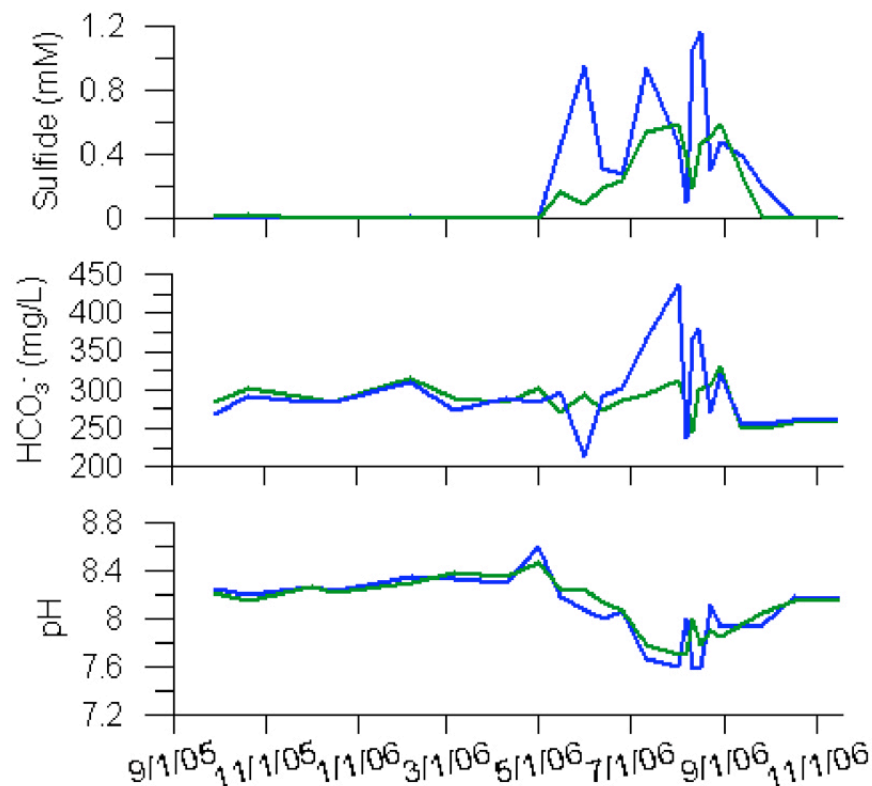


Figure 4. Sulfide and bicarbonate concentrations and pH in the bottom water of the north and south basins of the Salton Sea (sites 6-2 and 14-3).

Sediment Fluxes

The concentration gradients between the sediment pore water and overlying bottom water were used to calculate the fluxes of dissolved sulfide, bicarbonate, and ammonium to the water column. Rates were obtained using a modified form of Fick's first law of diffusion:

$$J = -\Phi^2 D_s (dC/dz),$$

where J is the diffusive flux in mass per unit area per unit time; Φ is the porosity ($\text{m}^3 \text{m}^{-3}$); D_s is the sediment diffusion coefficient, and dC/dz is the change in concentration with respect to a distance below the sediment-water interface (Wardlaw and Valentine 2005). A depth interval (dz) from 0 to 10 cm (average of 5 cm) was used to calculate the concentration gradient between pore water and bottom water.

The calculated fluxes of sulfide, bicarbonate, and ammonium for the center of the north south basins (sites 6-2 and 14-3) are shown in figure 5. A significant predicted flux was observed from the sediment to the overlying bottom water (*fig. 4*). The predicted rates of flux increased through the summer to maximum values, approaching $4 \text{ mmol m}^{-2} \text{ d}^{-1}$ at site 10-2, on September 28 (*fig. 4*). Predicted flux rates then slowed in the fall, presumably coinciding with reduced water column temperatures and higher DO levels (*fig. 2*).

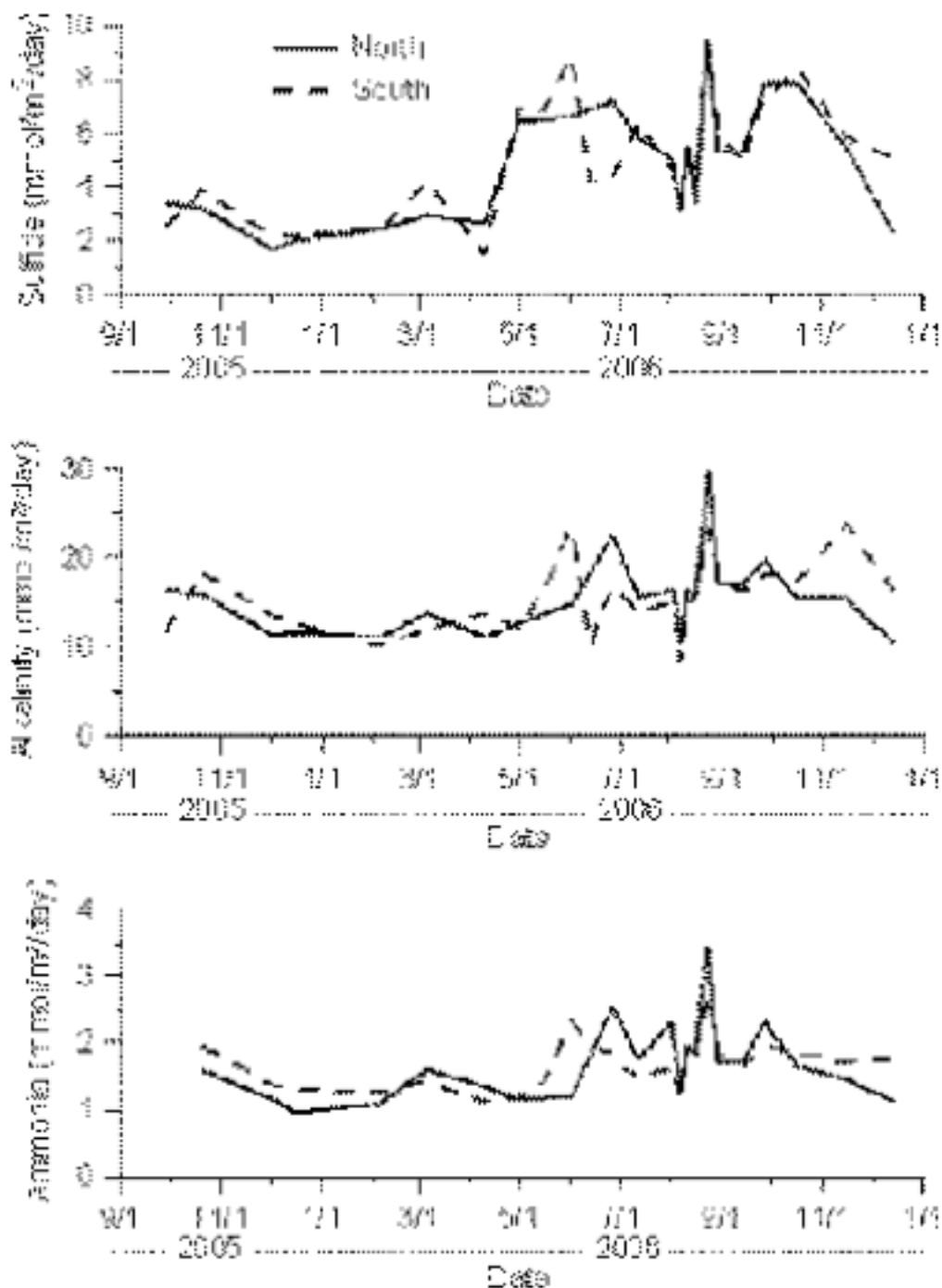


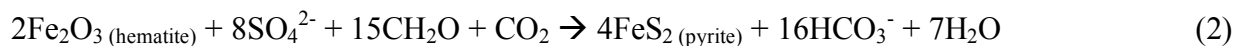
Figure 5. Flux of dissolved sulfide, alkalinity (HCO₃⁻), and ammonia (NH₃+NH₄⁺) from the sediment to the overlying water.

Iron Sulfide Mineral Precipitation

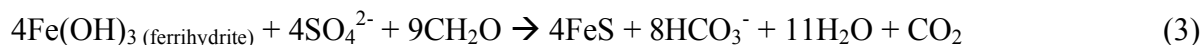
In spite of low redox potential in the sediments, Fe²⁺ was not present in any significant concentration in porewater from the upper 10 cm of sediment (<0.05 mg/L). The high levels of sulfide result in the precipitation of Fe²⁺ as insoluble iron monosulfides (FeS) and pyrite (FeS₂),

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thus limiting Fe^{2+} accumulation in the sediment porewater and lower water column. The concentrations of amorphous FeS, greigite (Fe_3S_4), pyrite, Fe(III)-oxides, and silicate-iron were determined on the sediments (Figure 6). The reductive dissolution of Fe(III)-oxides and the precipitation of pyrite or FeS also generates alkalinity, contributing to the production of “new alkalinity” and calcite precipitation:



If organic matter oxidation is coupled with ferrihydrite reductive dissolution and FeS precipitation, alkalinity is formed:



The concentrations of amorphous FeS and pyrite were highest in the south basin sediments due to the input of reducible Fe(III) from the river borne suspended solids from the New and Alamo Rivers. Approximately 88% of the measured suspended solids load to the Salton Sea enters through these two rivers (Holdren & Montaño (2002). Based on the concentrations of readily-reducible iron entering the Sea on river borne suspended solids, we estimate that 7,700 metric tons of pyrite are forming annually in the Sea (deKoff, et al. 2007).

Atmospheric Fluxes

The sulfide that diffuses from the sediments to the overlying lake water is either reoxidized to sulfate via reaction with dissolved oxygen or is lost to the atmosphere as $\text{H}_2\text{S}_{(g)}$ at the surface of the lake. While sulfate reduction is an alkalinity-generating reaction (eq 1), reoxidation of sulfide in the water column consumes alkalinity through the production of protons:



Thus, the reoxidation of H_2S and HS^- to SO_4^{2-} reduces the amount of alkalinity produced in the lake water. The net export of $\text{H}_2\text{S}_{(g)}$ from the basin is an important component for the permanent burial of carbon in the sediments. Export of H_2S from the Salton Sea and other closed agricultural basins can be achieved via volatilization from the water column to the atmosphere. As a result, atmospheric sampling for $\text{H}_2\text{S}_{(g)}$ (and $\text{NH}_3_{(g)}$) was conducted in concert with the water column and pore water sampling.

The concentration of H_2S in the air 1-2 m above the surface of the Salton Sea averaged about $1 \mu\text{g m}^{-3}$ through the winter and spring, and were consistently elevated compared to those of the background locations (*fig. 7*). For comparison, Robinson (1970) estimated the average ambient air level of hydrogen sulfide in urban areas to be $0.3 \mu\text{g m}^{-3}$. The California state-wide ambient air quality standard for H_2S is $42 \mu\text{g m}^{-3}$ (Collins and Lewis 2000).

The two-week average H_2S concentration at the target peaked at $35 \mu\text{g m}^{-3}$ during the period of the deep mixing event that began on 7/30/2006, which brought bottom water to the surface of the lake.

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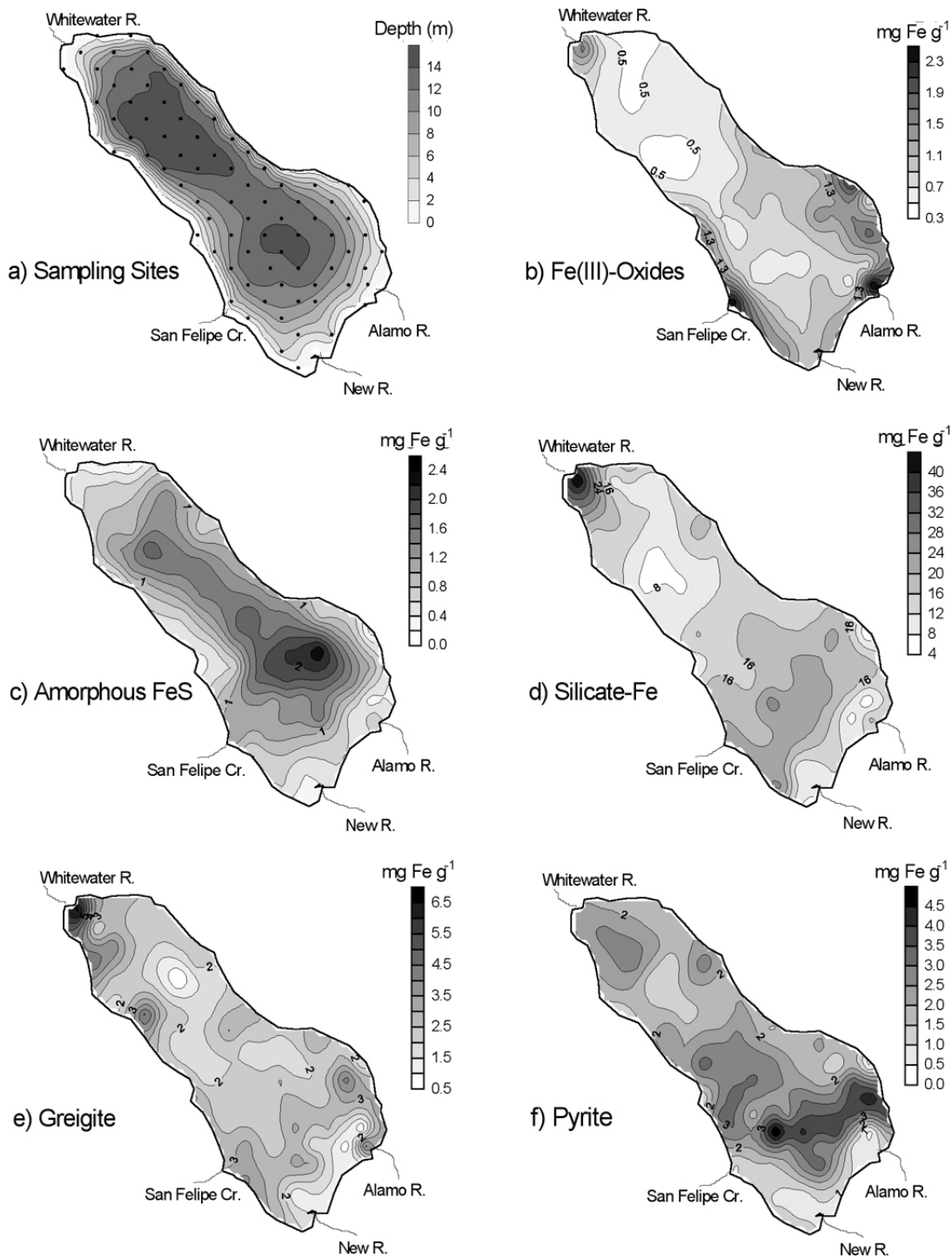


Figure 6. a) Bathymetry of the Salton Sea and sediment sampling locations used for iron extractions. Distribution of: b) Fe(III)-oxides, c) amorphous FeS, d) silicate iron, e) greigite (Fe_3S_4), and f) pyrite (FeS_2) in Salton Sea sediments.

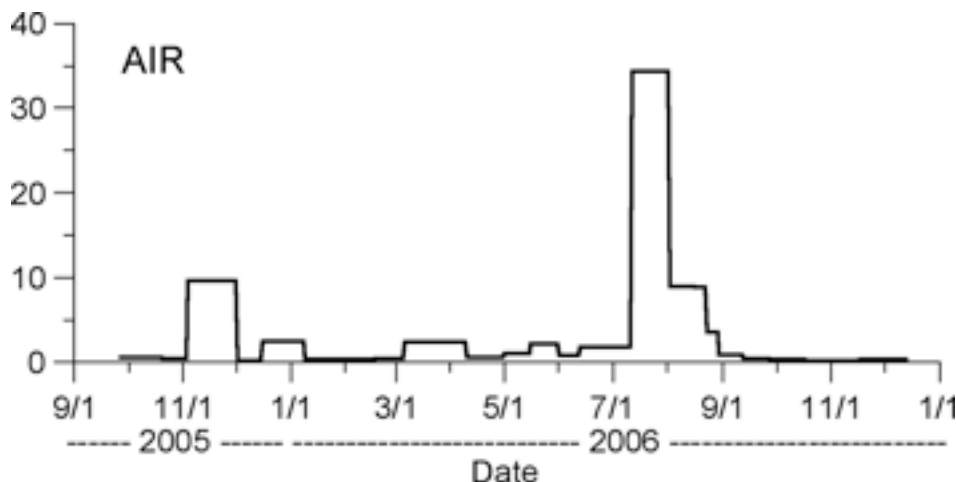


Figure 7. Atmospheric concentrations of H₂S gas at the “target.” Passive diffusion samplers were collected and replenished every two weeks on average.

The fluxes of hydrogen sulfide and ammonia at the air-water interface (*fig. 8*) were calculated using a modified Two-Layer Model (Liss and Slater 1974):

$$J = v_{aw}([H_2S]_w - \frac{[H_2S]_a}{K_H}) \quad (6)$$

where v_{aw} is the overall transfer velocity at the air-water interface, which takes into account the molecular diffusivity in the air phase (Fuller et al. 1966) and water phase (Hayduk and Laudie 1974). The diffusion coefficients were corrected for dynamic viscosity (Boudreau 1997), air temperature, wind speed at 10m above the surface, and atmospheric pressure. The concentrations in the water and air phases (denoted by subscripts w and a, respectively) were used for each sampling date. K_H is the non-dimensional Henry’s Law constant, which was corrected for salinity and temperature.

Alkalinity Fluxes from the Constructed Wetlands

There appeared to be a net flux of alkalinity out of the Brawley wetlands relative to chloride, which is considered a conservative tracer (*fig. 9a*), but the Imperial wetlands did not show this consistent production (*fig. 9b*). Based on additional analysis of the water quality data it appears that the variability of the sulfate, calcium, chloride, and bicarbonate concentrations in the inflow and outflow from these two wetlands, we conclude that sulfate reduction is not occurring to any significant extent. This is probably due to the low sulfate concentrations in the river and drain waters, and the high dissolved oxygen concentrations typical throughout the wetlands.

Discussion

The concentration gradients were used to estimate the fluxes of sulfide, alkalinity, calcium, and ammonium across the sediment-water interface. The fluxes were integrated over time and area summed for the sampling sites. The cumulative flux of sulfide (as S²⁻) from the sediments was 58,000 Mg y⁻¹ (metric tons per year). This mass of sulfide would generate sufficient bicarbonate to precipitate 136,000 Mg y⁻¹ of CaCO₃, assuming the H₂S/HS⁻ ratio is 1.0. However, most of

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this hydrogen sulfide is reoxidized in the water column so it cannot be counted towards the net accumulation of calcite.

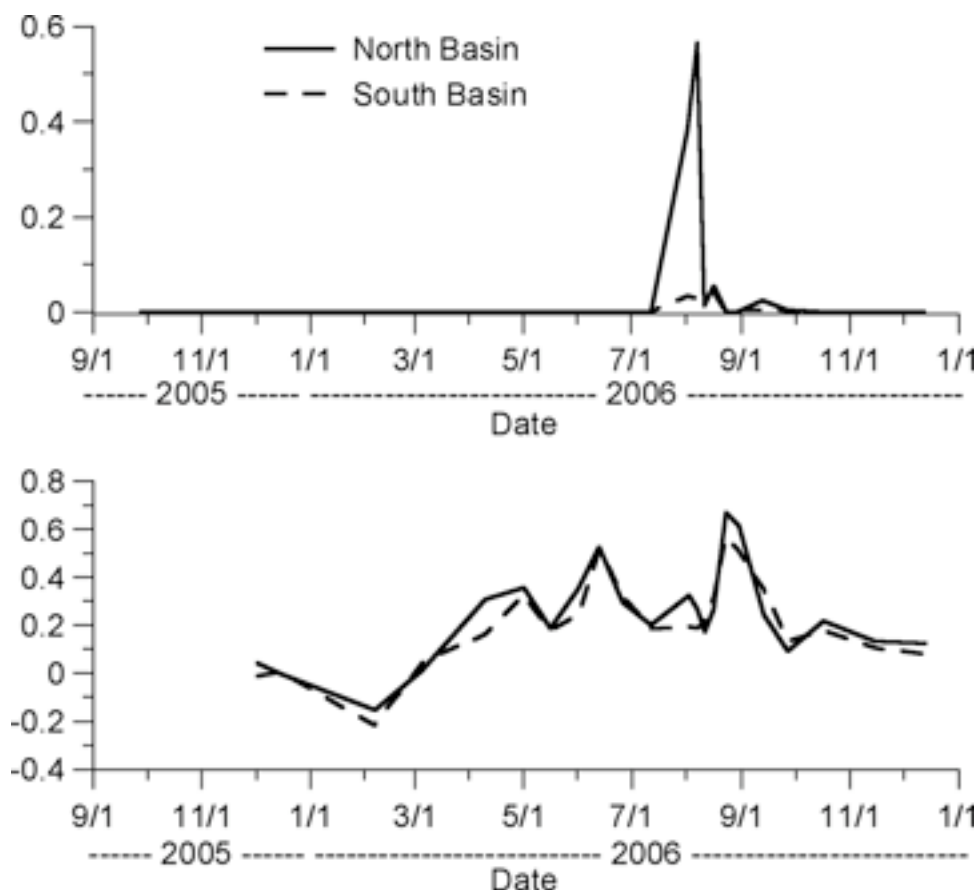
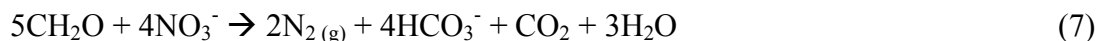


Figure 8. Fluxes of $\text{H}_2\text{S}_{(g)}$ and $\text{NH}_3_{(g)}$ across the air/lake surface interface.

The flux of calcium into the sediments results in the precipitation of $93,200 \text{ Mg y}^{-1} \text{ CaCO}_3$ within the sediments and the precipitation of pyrite produces another $12,800 \text{ Mg y}^{-1} \text{ CaCO}_3$. The bicarbonate flux out of the sediments is sufficient to precipitate $297,000 \text{ Mg y}^{-1} \text{ CaCO}_3$. Based on the average annual inflow of bicarbonate and calcium to the Sea we estimate with geochemical modeling (PHRQPITZ) that $340,000 \text{ Mg y}^{-1}$ of CaCO_3 would be forming in the absence of sulfate reduction. Thus, “new alkalinity” due to sulfate reduction appears to be a significant contributor to the total calcite precipitated.

The average annual input of nitrate-nitrogen to the Salton Sea is $8,800 \text{ Mg y}^{-1}$ (Holdren and Montañó (2002). If all of this nitrate is denitrified to N_2 gas via Eq. 7, then $3,100 \text{ Mg y}^{-1}$ of CaCO_3 could be produced from the bicarbonate formed.



The volatilization of H_2S was calculated to be 335 Mg y^{-1} . For comparison, the total annual California statewide industrial emissions of H_2S from facilities reporting under the Air Toxics Hot Spots Information and Assessment Act is $2,600 \text{ Mg y}^{-1}$ (Collins and Lewis 2000). Thus, the Salton Sea can be considered a significant source of H_2S and may be comparable to living next to an industrial emitter. The loss of H_2S gas from the lake is a permanent loss of acidity and generates sufficient alkalinity to precipitate $980 \text{ Mg y}^{-1} \text{ CaCO}_3$.

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The volatilization of ammonia reduces the alkalinity of the lake when ammonium (NH_4^+) dehydrates to ammonia (NH_3), leaving H^+ in the water. Potentially, every mole of NH_3 that volatilizes will consume one mole of HCO_3^- , converting the bicarbonate to CO_2 . The flux of ammonia from the lake was measured at $5,700 \text{ Mg y}^{-1}$. This loss of ammonia could reduce the net calcite precipitation by $33,500 \text{ Mg y}^{-1}$.

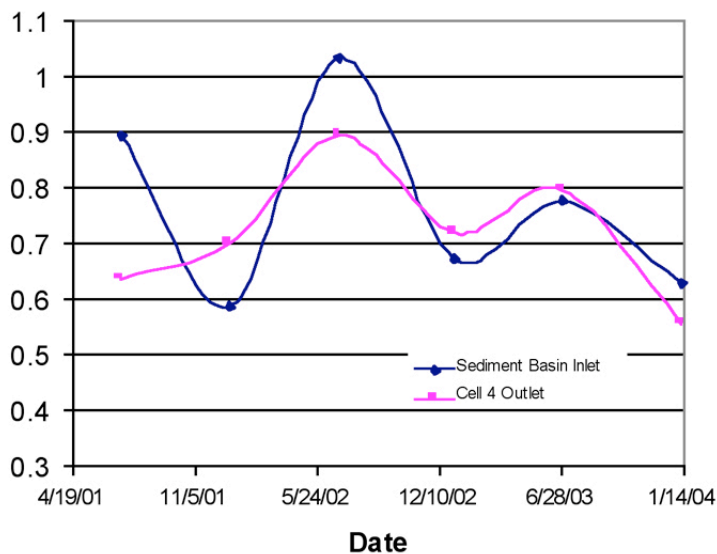
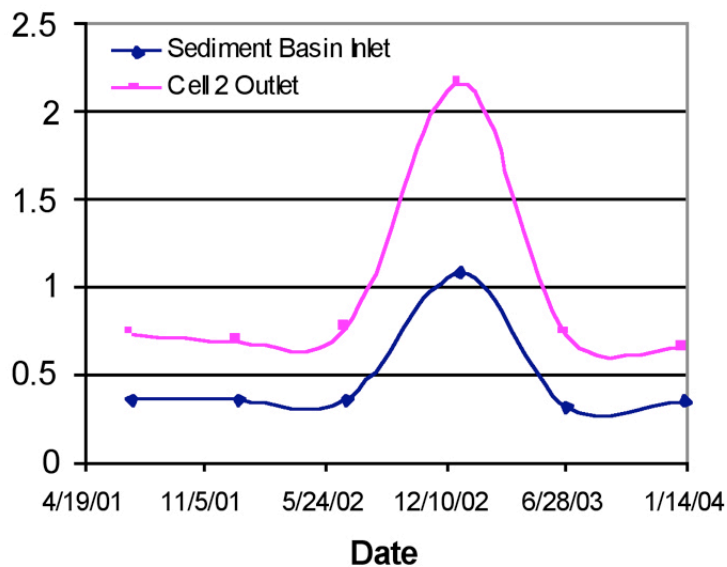


Figure 9. Changes in the HCO_3^- to Cl ratio for the inflow and outflow from the a) Brawley wetlands and, b) the Imperial wetlands.

The oxidation of sulfide in the water column releases protons, neutralizing alkalinity. Based on the pH of the pore water, the H_2S to HS^- ratio entering the water column is 1.0, which will produce 1.5 moles of H^+ per mole of sulfide, which is enough acid to neutralize 2,700 Mmol of HCO_3^- , or reduce calcite precipitation by $136,000 \text{ Mg y}^{-1}$.

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Summing all of these gains and losses of CaCO_3 due to anaerobic reactions, volatilization losses, and oxidation of sulfide yields a total of $238,000 \text{ Mg y}^{-1}$. Thus, the formation of “new alkalinity” increase the amount of calcium carbonate precipitating in the Salton Sea by 1.7 times and the estimated total calcium carbonate precipitation is $578,000 \text{ Mg y}^{-1}$.

An independent estimate of the amount of calcium lost from the Sea during the period 1980 to 1997 is $344,000 \text{ Mg y}^{-1}$ as Ca (Tostrud 1997). If all of this calcium had precipitated as CaCO_3 the total calcite formed would be $860,000 \text{ Mg y}^{-1}$. Therefore, the difference between $860,000 \text{ Mg y}^{-1}$ and $578,000 \text{ Mg y}^{-1}$ is the amount of calcium that is going to gypsum precipitation in the Sea. Geochemical modeling indicates that the Salton Sea is oversaturated with respect to gypsum, and gypsum crystals are recovered from the sediments, particularly in the shallow portions of the lake where anaerobic reactions are less intense. Thus, $485,000 \text{ Mg y}^{-1}$ of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) is forming in the Salton Sea.

The total mass of calcite precipitated in the soils of the Imperial and Coachella valleys has been estimated by several researchers over many years (Bower et al. 1969; Suarez and Rhoades 1977; Suarez 1999). Based on these studies, approximately $350,000 \text{ Mg y}^{-1}$ of calcite is precipitating within the soils of the Imperial and Coachella valley irrigation projects. Thus, the downstream reactions of calcite precipitation within the terminal basin of the Salton Sea far exceed the inorganic carbon storage within the soils of the valleys.

It is recognized that agriculture has had a global impact on carbon storage and cycling in soils. Much effort has been placed on quantifying the changes in organic carbon stored in soils and to a lesser extent, the changes in inorganic carbon (Lal 2001). In this study, we demonstrate that the discharge of agricultural wastewater to a terminal basin will result in carbon burial as calcite that can far exceed the amount of calcite precipitating within the soils. In addition, chemical reactions within the anaerobic parts of the basin convert nitrate and sulfate to bicarbonate, thereby increasing the net inorganic carbon burial. Downstream reactions within a closed irrigation basin need to be considered when considering the changes in carbon storage due to irrigated agriculture.

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