



# Variations in the release of silicate and orthophosphate along a salinity gradient: Do sediment composition and physical forcing have roles?



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## ABSTRACT

It was hypothesized that sediment composition, i.e. organic matters and minerals, and physical forcing can influence retention and release of silicate ( $\text{SiO}_4$ ) and orthophosphate ( $\text{o-PO}_4$ ) along salinity gradients. An experiment was performed to measure nutrient release by using treatments with and without sediment organic matter from the Guadalupe and Nueces Estuaries at five different salinities. The sample mixtures were shaken at intervals over the course of 48 h to simulate wind and river forcing. The release of silicate from sediments increased with time from 2 min to 48 h in all five salinities. The added orthophosphate concentration was adsorbed in most of the sediment containing organic matter and calcium-rich shells from both estuaries. From the sediments without organic matter, the release of orthophosphate was as high as  $52 \mu\text{mol/L}$ . The sediment minerals quartz and calcite were abundant in both estuaries. The average quartz to corundum peak intensities ratio were 14.04 and 13.36 and the average calcite to corundum peak intensities ratio were 3.06 and 1.32 in the Guadalupe and Nueces Estuaries respectively. The average organic matter in the Guadalupe and Nueces estuaries were 10.67% and 13.39% respectively. The retention and release of orthophosphate from the sediments may have been caused by the bonding with organic matter and calcite in the sediments. These findings indicate that sediment composition was a significant contributor in the low dissolved orthophosphate concentration in the estuaries. The release of silicate from the sediments containing quartz, and organic matter, when shaken, indicate that the combined forcing of river and wind may have been maintaining the estuaries silicate concentrations.

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## 1. Introduction

The fluctuation of silicate and phosphate concentrations along estuarine salinity gradients might be due to the interaction between sediment composition and overlying water (Morris et al., 1981; D'Elia et al., 1983; Anderson, 1986). The sediment composition includes sediment organic matter and minerals that interact in the presence of salt water electrolytes. This, in turn, may affect retention-release, precipitation-dissolution and flocculation-deflocculation of dissolved silicate and phosphate.

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According to previous studies, transported dissolved silicate decreased in concentration as it moved from a river to an estuary salinity gradient (Morris et al., 1981; Conley and Malone, 1992; Eyre and Balls, 1999). Different explanations have been suggested for the decrease in silicate along river-mouth to estuary-ocean salinity gradients. Those include coagulation of a colloidal form of silica (Krauskopf, 1956), retention of silicate on clay minerals as it moves from freshwater to the salt water interface (Williams and Crerar, 1985), and silicate uptake by diatoms (Conley and Malone, 1992). A sudden increase in silicate concentration in oligohaline (salinity of 0.5–5, Venice Classification system; Anonymous, 1958) regions from riverine freshwater concentrations may be a result of the dissolution of biogenic silica present in river water (Anderson, 1986).

Previous studies have revealed two types of phosphate behavior in estuarine salinity gradients. The first one is the decrease in

dissolved phosphate concentration from fresh to saline water gradients with most of the losses at the confluence of a river and an estuary. There are several potential causes of this sudden decrease in concentration; a change in pH (Garcia-Luque et al. 2006), precipitation of phosphate-colloid (Bale and Morris, 1981), and retention of phosphate onto suspended clay particles (Garcia-Luque et al. 2006). The second type of phosphate behavior was the increase in phosphate concentration in the oligohaline regions because of the release of phosphorus from the iron and aluminum oxide organic complex as the river water mixes with the estuary water (Sundareshwor and Morris, 1999). Flocculation of organic matter inhibits release from organic complexes as sediment is transported to mesohaline (salinity of 5–18, Venice Classification system; Anonymous, 1958) and polyhaline (salinity of 18–30, Venice Classification system; Anonymous, 1958) regions, and result in the decrease in phosphate concentration in the oceanic end of an estuary (Howarth et al., 1995; Nielson et al., 2001).

Recent research has found a salinity of 35 can dissolve silica gel and release silicate in the water (Tanaka and Takahashi, 2013). Additionally, Spagnoli and Bergamini (1997) identified the release of phosphate during resuspension. The interaction between sediment composition and water in the estuaries could be important in the retention and release of silicate and orthophosphate from the sediment. A weekly nutrient study in Nueces and Corpus Christi Bay identified very low orthophosphate ( $\text{o-PO}_4$ ) concentrations (mean = 0.12  $\mu\text{mol/L}$ ), and high silicate ( $\text{SiO}_4$ ) concentration (mean = 127.5  $\mu\text{mol/L}$ ) in all samplings (Turner, 2014). Phosphate can adsorb onto clay particles resulting in reduced orthophosphate concentration (Garcia-Luque et al. 2006), the low orthophosphate concentration might be because of the absorption onto clay particles. Furthermore, in the Guadalupe and Nueces estuaries salinity and resuspension induced by wind or river flow may be the major factors that cause variation in sediment nutrient release. Therefore it is important to identify the effects of estuarine sediment composition at various salinities and the retention and release of  $\text{SiO}_4$  and  $\text{o-PO}_4$  concentrations due to resuspension. Thus, the present study is to characterize silicate and orthophosphate release from sediment in a laboratory setting by varying sediment organic matter, salinity and agitation rate. Agitation was used to mimic river flow and wind forcing.

## 2. Materials and methods

### 2.1. Sediment sample collection sites and bottom water quality

The top 5 cm of sediment were collected using cores from station 'A' of Guadalupe and Nueces Estuaries during July 2012, October 2012, and January 2013 quarterly samplings (Fig. 1). Data collected from station A of both estuaries was used because it was closer to the river mouth, i.e. near to the head of the estuaries, and was therefore assumed to have higher organic loads. In the field, sediment samples were kept on ice and stored frozen upon transfer to the lab in order to slow organic matter degradation. Organic contents, mineral contents and nutrient release experiments were performed by using these sediment samples.

Temperature, pH, salinity, dissolved oxygen (DO), were collected using YSI sonde in the bottom water (20 cm above the sediment surface) of the two estuaries. Water samples were also collected for nutrient analysis.

### 2.2. Sediment analysis and laboratory experiments

The laboratory experiment was conducted at room temperature. Before the sediment was used for laboratory experiment, organic matter, and mineral analyses it was carefully homogenized.

#### 2.2.1. Organic matter

The organic matter content in the sediment sample was determined by  $\text{H}_2\text{O}_2$  digestion (Schumacher, 2002).  $\text{H}_2\text{O}_2$  is an oxidant; its addition oxidizes organic matter and decreases the pH (up to 3–4). The decrease in pH could dissolve calcite and calcium carbonate shells that can release calcite bonded phosphorus (Staudinger et al., 1990). Care was taken to avoid excessive frothing and dissolution of calcite mineral. Our main goal on using  $\text{H}_2\text{O}_2$  was to digest organic matter and observe nutrient release from sediments.

A 10–15 g sediment sample was weighed and dried overnight at 60° C in an aluminum pan. After drying, the samples were reweighed and each placed in a beaker containing 50 ml of 30%  $\text{H}_2\text{O}_2$ . The sediments from both estuaries contained clam and oyster shells, which might get corroded by  $\text{H}_2\text{O}_2$  solution. Effort was made to preserve sediment characteristics by not keeping the sediment for too long in the solution. After the digestion of organic matter the supernatant was decanted and an aliquot of deionized (DI) water was added. The beaker was stirred and allowed to settle until the supernatant was clear. This process was repeated two more times. The sample was then filtered by pouring the beaker contents through a 0.7  $\mu\text{m}$  filter paper suspended in a glass funnel. The sediment collected on the filter paper was oven dried at 60° C overnight. The samples were again weighed. Sediment organic matter was calculated by the difference in weight before and after organic matter digestion. From here on samples digested with  $\text{H}_2\text{O}_2$  refers to without organic matter treatment.

#### 2.2.2. Mineral content

Sediment mineral has a role in fluctuating inorganic nutrient concentration by retention and release into the mineral surface. The sediment without organic matter was ground using a mortar and pestle, and 2 g of sample was mixed with 0.5 g of corundum ( $\alpha\text{-Al}_2\text{O}_3$ ) in 4:1 ratio (Naehr et al., 2000). A few drops of ethyl alcohol were also added to the sample-corundum mixture to help in the homogenization. The prepared sample was then placed in a sample holder of a Rigakut Ultima III X-ray diffractometer and sediment mineral content was identified. In the x-ray diffractometer, scans were run from 2° to 80° 2 $\theta$  with scanning speed of 0.01° 2 $\theta$ /s. The crystal structure of each mineral is composed of sets of planes responsible for 'd' values (distance between array of crystal in mineral), and which is responsible for the variations in the peak. The peak, obtained as intensity count per second of the mineral type was compared with the corundum peak, and the mineral content was identified based on the mineral to corundum ratio. MDI Jade – 7 software (Bishop et al., 2011) was used to identify mineral types and peak intensities. The software can automatically identify the mineral content in the sample based on the location of the peak. However, the user has to differentiate the mineral type based on the 'd' value and one main peak of the mineral.

#### 2.2.3. Preparation of nutrients spike solution

Primary standards concentration of 10,000  $\mu\text{mol/L}$  were prepared using 1.36 g of potassium phosphate monobasic ( $\text{KH}_2\text{PO}_4$ ) and undried 2.12 g of sodium metasilicate pentahydrate ( $\text{Na}_2\text{SiO}_3$ ). Each chemical was weighed out, placed in 1000 ml volumetric flask, dissolved and brought up to 1000 ml with freshly drawn Milli-Q water. From these refrigerated primary standards, a 100 ml mixed stock standard was prepared daily containing 500  $\mu\text{mol/L}$   $\text{o-PO}_4$  and 5000  $\mu\text{mol/L}$   $\text{SiO}_4$ . That solution was then used to 'spike' the solute with additional nutrients in the following experiments.

#### 2.2.4. Experimental design for laboratory study

All the laboratory experiments to identify sediment nutrient release were performed within two weeks of sample collection. The

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