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Baseline

Bioavailability and geochemical speciation of phosphorus in surface sediments of the Southern Caspian Sea



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ABSTRACT

The purpose of this study was to quantify phosphorus bioavailability and various forms of this element in surface sediments of the southern Caspian Sea. To do, sediment samples were collected from different depths in autumn 2015 using a Van Veen Grab. Phosphorus forms were recognized by a sequential extraction procedure (SEDEX). Results revealed that the range of total phosphorus (TP) was between 431 ppm and 594 ppm with a mean value of 535.25 \pm 45.05 ppm. Organic phosphorus (OP) ranged from 62 to 99 ppm and contributed 14.85 \pm 2.21% of total phosphorus in average. The contents of loosely phosphorus, iron-bound phosphorus, authigenic phosphorus and detrital phosphorus varied 35–55 ppm, 50–94 ppm, 125–189 ppm and 152–217 ppm, respectively. Generally, inorganic phosphorus (IP) was between 365 and 522 ppm which comprised 81.9–88.53% of total phosphorus. Findings showed levels of different phosphorus forms in a descending order: detrital P > Authigenic P > Organic P > Fe-P > Loosely P. Bioavailable phosphorus concentration was between 153 ppm and 240 ppm and consisted 37.21 \pm 3% of total phosphorus. Based on molar ratio of TOC/OP, organic matter obtained from the studied stations suggested a terrestrial origin.

Phosphorus as a substantial element, plays a crucial role in the regulation of bio-community structure and biogeochemical cycle of other elements in an aqueous environment. Phosphorus is regarded as a part of those elements forming DNA and ATP (Lehninger et al., 1993; Tyrrell, 1999; Ruttenberg, 2003; Reddy et al., 2005; Paytan and McLaughlin, 2010), many other cellular components like phosphoprotein and phospholipid in cell membrane, teeth and bones. Phosphorus availability in aqueous systems affects primary production rate, species distribution and ecosystem structure (Karl et al., 2001; Paytan and McLaughlin, 2010). In a large number of marine and estuarine environments, phosphorus availability acts as a limiting factor in primary production (Smith, 1984).

Phosphorus in oceanic and marine waters is usually released by rock weathering and erosion of the continents. This phosphorus is discharged through rivers into aqua ecosystems either in dissolved form or with suspended materials. Additionally, other factors such as deposition of suspended particles in the atmosphere, volcanic ash and inorganic dust are also important in increased phosphorus loadings into the environment (Delaney, 1998; Benitez-Nelson, 2000). Two forms of suspended phosphorus in river flows are organic and inorganic phosphates of which the latter is mostly common in rivers, especially the phosphorus in apatite and other minerals, phosphorus adsorbed by the surface of oxide compounds and ferromagnesian hydroxides. This part of phosphorus in estuaries and coastal sediments is deposited and not directly usable by living organisms (Wollast, 1983).

However, clay minerals along with ferrous and aluminum oxyhydroxides have high capacity for orthophosphate in aqua environments. After being transported to estuaries where salinity changes, these particles are separated from the clay and enter to marine waters. In several studies, it has been estimated that the amount of orthophosphate discharged into the seas and oceans by resorption process from the surface of clay minerals is approximately two to five fold greater

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than that is released through rivers (Sundareshwar and Morris, 1999).

In marine waters, phosphorus is found either in dissolved or suspended forms, each have both inorganic form (orthophosphate, pyrophosphate, polyphosphate and phosphate in minerals) and organic form (mono- and diester, phosphonite). Organic and inorganic forms of phosphorus suffer a continuous transformation. Dissolved inorganic phosphorus (usually as orthophosphate) is absorbed by phytoplankton and changed into organic phosphorus compounds (Frankowski et al., 2002; Aydin et al., 2009).

In sediments, phosphorus is discovered as Loosely-P, Fe-bound, Authigenic-P, Organic-P and Detrital-P. Hence, identification of phosphorus forms in aqua environment is of great importance (Ruttenberg, 1992). Various studies have been conducted on different forms of phosphorus in several regions so far (Liu et al., 2004; Reddy et al., 2005; Kraal et al., 2012; Yu et al., 2013; Meng et al., 2014), but little information is known in the Caspian Sea. Therefore, the present work aimed to identify different forms of phosphorus in the Caspian Sea sediments and their relations with environmental factors.

Surface-sediment sampling was conducted at 12 stations along four transects (3 stations at each transect) using a Van Veen Grab in autumn 2015. Geographical location of the sampling points in the southern Caspian Sea is illustrated in Fig. 1. Samples were carried to the laboratory and then kept at - 20 °C prior to analyses. For chemical differentiation of phosphorus, sediments were firstly sieved through a mesh-size screen of $63 \,\mu\text{m}$ and then $0.5 \,g$ freeze dried sediment of each sample in 50 ml extractant were analyzed using Ruttenberg method (Ruttenberg, 1992). Excess use of solids can lead to incomplete dissolution of target phases because the solution would saturate relating to that target phase, therefore, the solid solution ratio has a crucial role in this speciation method (Ruttenberg, 1990). In Fig. 2, sequential extraction procedure is displayed in details, by which phosphorus is classified as different forms of loosely-P, iron-bound P, authigenic-P, detrital-P and organic-P. In addition, sediment sample used in a stage was then applied for the next stage. It is worth noting that the extracted solution was filtered through a 0.45 µm GF/C filter membrane before phosphorus measurement and pH of extract was then reached to neutral point (Zhang et al., 2010; Lin et al., 2013). In each phase and after extraction, samples were centrifuged at 4000 rpm for 20 min and phosphorus content was measured according to the method of molybdenum blue/ascorbic acid (Murphy and Riley, 1962) at 880 nm wavelength using a UV-VIS spectrophotometer. Inorganic-P included loosely-P, iron-bound P, authigenic-P and detrital-P altogether. All the tools applied in digestion process and phosphorus assessment were placed in 5% nitric acid for 24 h and finally rinsed with twice distilled water before using. Total organic carbon (TOC) was measured by a TOC analyzer (SGE, ANATOC Seri II Australia). For particle size analysis,



Step Extractant Extracted



Fig. 2. Procedures used for sequential extraction of different forms of sedimentary phosphorus (Ruttenberg, 1992).

Sodium hexametaphosphate was added to a proportion of dried sediment samples. Thereafter, sand and mud determination was performed by a laser particle analyzer (HORIBA-LA950, France & Japan).

Data normality was checked by Kolmogorov–Smirnov test. Mean comparison was conducted via Duncan's multiple range test after data were subjected to one-way ANOVA. Pearson correlation test was used to investigate the relations among measured parameters. Statistical analysis was performed using SPSS software Ver. 18. Multivariate analysis including principle component analysis (PCA) and cluster analysis (CA) were employed using Primer 6 & PERMANOVA software. Cluster analysis, as a mathematical approach determines the similarity

Fig. 1. The locations of sampling sites at the southern Caspian Sea.

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