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硕士学位论文

**DGT 的制备、性能测试及  
在海水分析中的应用**

**Preparation, Characterization and Application of DGT  
for Analysis of Trace Metals in Seawater**

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## 摘 要

重金属在海水中以各种化学形态存在。研究表明与生物体直接作用的并不是总溶解态金属，而是溶解态金属中的一部分，这部分被定义为活性 (labile) 金属。海水中重金属的存在形态是研究其毒性和生物可利用性的关键参数。

目前研究海水中重金属的形态，一般需将样品采集回实验室后进行分析。但水样中重金属的化学形态容易在样品采集、运输、预处理与保存过程中发生转化，且各种试剂的添加也会对重金属的化学形态分布造成影响，因此原位富集技术的开发应用是十分必要的。

薄膜扩散梯度技术(Diffusive Gradients in Thin-films Technique, DGT)是 1994 年提出的一种原位富集有效态重金属的新技术。DGT 采样装置的关键部件包括扩散相和结合相；水体中的重金属通过扩散相进入结合相而被富集，富集量与时间有关而与样品体积无关。

本研究制作了三种结合相。一是广泛采用的 Chelex-100 树脂凝胶，二是戊二醛改性壳聚糖膜，三是戊二醛-L-半胱氨酸-壳聚糖膜。采用改性琼脂糖交联的聚丙烯酰胺凝胶作为扩散相。自制 DGT 采样装置，对其进行性能测试，并应用于海水中有效态重金属的原位富集。主要研究内容及结果如下：

(1) 使用改性琼脂糖交联的聚丙烯酰胺凝胶作为扩散相，并在凝胶中嵌入 Chelex-100 树脂作为结合相。应用该 DGT 采样装置同时原位富集海水中的锰、钴、镍、铜、锌、镉、铅。使用自制的凝胶扩散系数测定装置测定了金属离子在该扩散相中的扩散系数。考察了 pH、高盐基底等对富集效果的影响。pH 在 5.6~8.6，离子强度在 10~700 mmol L<sup>-1</sup> 范围内，富集效果良好。在厦门近岸海域成功地进行为期 7 d 的海水中重金属的原位富集实验，结果显示，DGT 能够富集该海域海水中溶解态重金属的 6.67%~45.33%，即获得 DGT 有效态。平行样测定的 RSD 均小于 10%。本方法能准确原位地富集海水中的生物有效态重金属，具有原位、便捷、pH 适用范围广、在河口和近海富集时基本不受离子强度影响及样品存放时间长等优点，为海水中痕量有效态重金属的分析提供了有效的采样技术。

(2) 在上述工作基础上建立了以改性琼脂糖交联的聚丙烯酰胺凝胶为扩散

相、以戊二醛改性壳聚糖膜为结合相的 DGT 采样装置。应用该装置同时富集加标海水中的钴、镍、铜、锌、镉。考察了戊二醛改性壳聚糖对不同浓度重金属的吸附效率,结果显示,在相同重金属浓度下,戊二醛改性壳聚糖膜的吸附效率高于 Chelex-100 树脂。考察了 pH、高盐基底等对富集效果的影响。结果显示, pH 在 5~8 范围内富集效果基本不受影响。离子强度为 10 和 700 mmol L<sup>-1</sup> 时,富集效果差别不大,说明该方法适用于河口及近岸海水中痕量重金属的分析。在实验室内成功地进行为期 3 d 的加标海水中重金属的原位富集实验,结果显示, DGT 能够富集海水中溶解态重金属的 12.57%~64.68%。平行样测定的 RSD 均小于 10%。与 Chelex-100 为结合相的 DGT 富集效果比较无显著差异,但戊二醛改性壳聚糖膜的制备过程较 Chelex-100 树脂凝胶的制备过程更简易。

(3) 在上述工作基础上,建立了以改性琼脂糖交联的聚丙烯酰胺凝胶为扩散相、以戊二醛-L-半胱氨酸-壳聚糖膜为结合相的 DGT 采样装置。应用该装置同时富集加标海水中的钴、镍、铜、锌、镉。实验结果显示,在相同浓度的重金属溶液中,戊二醛-L-半胱氨酸-壳聚糖膜吸附效率高于 Chelex-100 树脂及戊二醛改性壳聚糖膜。pH 在 5~8 范围内富集效果基本不变。测定离子强度分别为 10 和 700 mmol L<sup>-1</sup> 时 DGT 的富集效果,结果显示两种离子强度下富集效果无明显差异,说明该方法适用于海水中痕量重金属的分析。采用该 DGT 采样装置,持续 3 d 采集加标海水中的重金属,获知该海水样品中 DGT 有效态重金属为溶解态重金属的 26.06%~87.25%,平行样的 RSD 均小于 10%。相同实验条件下,以戊二醛-L-半胱氨酸-壳聚糖膜为结合相的 DGT 装置所富集的有效态比例,高于以 Chelex-100 树脂凝胶及戊二醛改性壳聚糖膜为结合相的 DGT。

**关键词:** 薄膜扩散梯度技术; 海水中重金属; 原位富集; Chelex-100 树脂; 壳聚糖

**ABSTRACT**

Heavy metals exist as variety of chemical species in seawater. Nowadays scientists are increasingly recognizing that it is the bioavailability of heavy metals that controls the potential of adverse effects rather than the total or filterable concentrations. Knowledge of the concentrations and chemical speciation of trace metals is essential to understand influence of those metals on marine organism.

To do the speciation analysis of heavy metals in sweater, the collected samples are ordinarily shipped back to laboratory for determination. The heavy metal species could be changed during transportation, pretreatment and preservation. The chemical reagents added might also change the distribution of chemical species. Therefore, it is necessary to develop *in situ* collection methods of heavy metal species in natural waters.

Diffusive gradients in thin films (DGT) is a recently developed technique, which is capable of *in situ* enrichment of labile metal species in aqueous systems for quantitative measurement. The key components of DGT device are ion-exchange resin and diffusive gel. Metals transport through the diffusion gel in a controlled way, which makes it possible to explore the concentration of labile species over the deploying time.

In this study classical polyacrylamide hydrogel was used as the diffusion gel, and three ion-exchange sorbents were chosen to develop the DGT devices. Those were Chelex-100 resin impregnated with polyacrylamide, glutaraldehyde-crosslinked chitosan membrane and glutaraldehyde-crosslinked L-cysteine modified chitosan membrane. The DGT devices had been characterized and deployed for enrichment of heavy metals in seawater. The main contents and results of the study are as follows:

(1) The DGT device consisted of a polyacrylamide diffusive gel and a Chelex-100 resin impregnated with polyacrylamide was built for the measurement of Mn, Co, Ni, Cu, Zn, Cd and Pb in seawaters. The molecular diffusion coefficient  $D$  of various metals through the polyacrylamide gel had been determined. With Chelex-100 resin as the ion-exchange resin, pH in the range of 5.6~8.6 would not influence the enrichment.



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