



CONCORDANT ^{210}Pb AND ^{137}Cs AGES OF BLACK SEA ANOXIC UNCONSOLIDATED SEDIMENTS

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Received 13 November 2009

Accepted 27 October 2010

Abstract: Two independent geochronologic radiometric methods: ^{137}Cs and ^{210}Pb were used to determine the sedimentation rate and subsequently to date the oldest sediments of a 50 cm core containing unconsolidated sediments collected at a depth of 600 m below sea surface in the anoxic zone of the Western Black Sea, off the Romanian town of Constanta. Both methods gave coincident value within experimental uncertainties of $0.42 \pm 0.20 \text{ mm} \cdot \text{y}^{-1}$ for ^{137}Cs and $0.49 \pm 0.03 \text{ mm} \cdot \text{y}^{-1}$ in the case of ^{210}Pb , within the Constant Initial Concentration model, which gave for the lower most sediments an age of $1.00 \pm 0.01 \text{ ka}$. The constant sedimentation model as well as the possible causes of observed discrepancy between experimental uncertainties are discussed.

Keywords: Black Sea, anoxic sediments, ^{137}Cs , ^{210}Pb , absolute age, sedimentation rate.

1. INTRODUCTION

The Black Sea, a large inland sea with an area of 422,000 km² and a maximum depth of 2212 m, communicates in its southwestern corner with the Mediterranean Sea by Bosphorus and Dardanelles, as well as in North with the Sea of Azov by the strait of Kerch with a net inflow of freshwater from central and middle-eastern Europe totaling 320 km³/year and a net inflow of 200 km³/year of seawater through the Bosphorus. For this reason, the salinity of the Black Sea varies between 14‰ and 19‰ and, at the same time, the oxygenated water representing a thin cover of about 120-180 m lying over a huge volume of salt water saturated with hydrogen sulfide making the Black Sea the largest anoxic marine basin (Murray *et al.*, 1989; Özsoy and Unluata, 1997).

In this way, at depth under the oxygenated to anoxic transition interface (pycnocline) there are no living organisms except extremophile bacteria able to use sulfate

(SO_4^{2-}) for oxidation of organic material and producing hydrogen sulfide (H_2S) and carbon dioxide. Therefore, the total absence of living organisms able to produce bioturbation, represents ideal conditions to preserve the sediment's structure for a very long time, and thus allowing the assessment materials on the seafloor, environmental pollution studies and intercalibration of tracers to determine the timing of event sequences (Robbins *et al.*, 1978; Carroll and Lerche, 2003; Dului *et al.*, 2009). As an absolute geochronology is indispensable, environmental radionuclides such as artificial ^{137}Cs and natural ^{210}Pb are very useful in measuring sediments movement and accumulation, thus providing direct and reliable estimation of depositional and postdepositional processes during the last 150 y (Goldberg, 1963; Krishnaswami *et al.*, 1971; Robins and Eddington, 1975; Robbins *et al.*, 1978; Appleby and Oldfield, 1983; Albrecht *et al.*, 1998; Appleby 2000, 2001; Abril 2004; Tylmann, 2004).

Although ^{210}Pb ages are limited to 150 y, as long as other independent measurements such as digital radiography (Iovea *et al.*, 2005), could confirm a nearly con-

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stant sedimentation rate for an extended period of time, whose numerical value deduced, by ^{137}Cs and ^{210}Pb methods, could be used to infer sediments ages greater than this threshold.

Previous measurements concerning ^{137}Cs and ^{210}Pb vertical distribution in the seafloor sediments of the Black Sea basin showed a variable sedimentation rate, rapidly decreasing from 17-20 mm/y near the mouth of the main Black Sea western tributaries: Danube, Dniestr or Dniepr rivers to 1 mm/y and less in the deeper and anoxic part of the basin (water depth greater than 200 m). In this zone, which encompasses more than 90% of the Black Sea surface, the total absence of bioturbation determined an extremely limited postdepositional mixing of sediments thus making ^{137}Cs and ^{210}Pb vertical distribution well preserved. This fact enables us to consider the sediments from anoxic zones as appropriate media to preserve a long term history of environmental events (IAEA, 2004; Ayçik *et al.*, 2004).

While ^{210}Pb accumulates continuously and its vertical profile reflects the events of the past 150 y, ^{137}Cs is an episode marker, used mainly to establish the 1964 maximum of atmospheric nuclear tests or the more recent 1986 Chernobyl accident (Penington *et al.*, 1973; Appleby, 2000). At the same time, it must be kept in mind that in sediments, Chernobyl ^{137}Cs was deposited in a single event, while ^{210}Pb is deposited continuously, so that the vertical profile of its specific activity could provide a more reliable absolute geochronology. In this case, radio-caesium is usually used to validate the ^{210}Pb ages. At present time, the existing HPGe gamma spectrometers, provided with carbon-epoxy windows, permit a simultaneous accurate determination of the specific activity of both radionuclides for the same samples with high accuracy. For this reason, a synchronized investigation of their vertical profile is highly recommendable as it could furnish a significant better age model and hence an improved absolute geochronology of the past century and half.

A correct estimation of sedimentation rate in the case of unconsolidated sediments whose accumulation, as inferred by other methods, could be considered as stationary for a long period of time, is very useful in dating, by extrapolation, the sediments older than maximum age limit of ^{210}Pb . This is the case of deep sea anoxic zones which include also the anoxic Black Sea basin, where the lack of bioturbation and a homogenous hydrodynamic environment, offer good conditions for a long term preservation of sediments. These circumstances, reflected by a constant sedimentation rate for time intervals of ka and more, make possible to estimate the ages of sediments older than 150 years.

Once a recent geochronology established, the analysis of vertical profile of different mineral fractions, as well as trace elements, could bring reliable data concerning the past events as reflected in sediments composition.

Consequently, in our study we have investigated sediments collected at a water depth of 600 m below sea level from an anoxic zone of the Western Black Sea basin which offers ideal conditions to study the depositional processes of the sediments. By taking into account that the radiographic images of sediments (Duliu *et al.*, 2009) point toward a stationary depositional process, in interpreting our radiometric data, we have presumed a constant sedimentation rate. This research, whose results concerning ^{137}Cs and ^{210}Pb absolute geochronology are the object of present paper, was done within the Romanian CLIMARISC multi-annual scientific project devoted to the investigation of the history of the Western Black Sea environment (Climarisc, 2010).

2. MATERIALS AND METHODS

Sediments

The investigated sediments were collected from the anoxic zone of the Black Sea during the June 2004 scientific cruise of the R/V *Mare Nigrum*, in a point located at 44°30' N, 30°45' E, at a depth of 600 m. Immediately after collection, the 12 cm diameter and 50 cm length BS 600 core was stored in vertical position in a refrigerator and then examined by means of a Siemens Somatom HQ Computer Tomograph. The core radiography showed the existence of about 250, horizontal, one millimeter thickness and almost parallel laminae of coccolithic ooze and coccolithic mud, undisturbed by biotic activity (Duliu *et al.*, 2009). Moreover, the same radiographic image showed a decrease of the optical density towards bottom of the core of less than 8%, attesting an almost constant degree of compaction and thus allowing us to extrapolate for the entire core the accumulation rate deduced for the upper layer of sediments by means of ^{137}Cs and ^{210}Pb geochronology.

Then, the core was longitudinally spitted into two halves, one of them being divided into 45 segments for further investigations. Each segment was then dehydrated at 105°C, homogenized and divided in four aliquots for further measurements, from which subsamples of 3 to 100 g were used for further radiometric assay.

Gamma spectrometry

^{137}Cs specific activity was determined twice, once at the Environmental Protection Agency – Arad, and the second time the National Institute for Physics and Nuclear Engineering – Horia Hulubei (Bucharest), while ^{210}Pb specific activity was determined only at the Environmental Protection Agency – Arad. In both cases, samples were placed in plastic boxes for 30 days to reach the radioactive equilibrium. In the Environmental Protection Agency – Arad laboratory, the gamma spectra of sediment samples weighting between 3 and 8 g, were measured using a CANBERRA HPGe detector provided with a carbon epoxy window, with a resolution of 1,8 keV at

^{60}Co 1332.5 keV line and approximately 50% relative efficiency. The spectra were recorded using a Canberra MCA 1000 multichannel analyzer. Total ^{210}Pb activity was measured by using the 46.5 keV line while the activity of supported ^{210}Pb was estimated by measuring ^{226}Ra content. Consequently, ^{226}Ra activity was measured using the 295.2 keV and 351.9 keV lines of ^{214}Pb and 609.3 keV line of ^{214}Bi by assuming the existence of radioactive equilibrium. Energy calibration was made using a mixed source (^{109}Cd , ^{139}Ce , ^{57}Co , ^{60}Co , ^{137}Cs , ^{113}Sn , ^{85}Sr , ^{88}Y , ^{203}Hg). The absolute efficiency of the detector was calculated by using the specific Canberra Genie2000, ISOCS software. In these conditions the total relative errors regarding ^{210}Pb specific activities varied between 8 and 20%.

In the National Institute for Physics and Nuclear Engineering-Bucharest laboratory, ^{137}Cs activity was determined by using a 10 cm thick lead shielded HPGe detector provided with an aluminum window, with a FWHM of 1.9 keV at ^{60}Co 1332.5 keV and a relative efficiency of 30% connected to an 8192 channels CANBERRA MCA by means of an AccuSpec A board. The total acquisition time was of 200000 s for samples weighting between 25 and 100 g. The analysis of recorded spectra was carried out with an OS2/Gennie PC Software. The spectrometric chain was calibrated with standard gamma source consisting of a mixture of ^{152}Eu and ^{137}Cs in the same geometry as the samples. Standard material MAPEP-02-S9 (Mixed Analyte Performance Evaluation Program-United States) was used to check the measurements accuracy. The relative errors in the case of ^{137}Cs specific activities varied between 5 and 13%. The presence of an aluminum window did not provide good conditions for observing the 46.5 keV line of ^{210}Pb .

^{210}Pb dating models

At present time, two models for calculating recent absolute ages from ^{210}Pb sediment vertical distribution are in use (Robbins *et al.*, 1978).

One of them, the Constant Initial Concentration (CIC) model assumes that: i) the ^{210}Pb accumulates at the sediment-water interface with constant rate (Crozaz *et al.*, 1964, Krishnaswami *et al.*, 1971; Appleby and Oldfield, 1978), ii) the supply of ^{210}Pb (depending on incoming flux of ^{210}Pb) in the sediment vary directly in proportion to the sedimentation rate, iii) the activity of ^{210}Pb supported by ^{226}Ra the sediments is independent of depth, iv) there is no post-depositional migration of the radionuclide within the sediments. If the supply of ^{210}Pb is constant and the sedimentation rate is constant, an increase of ^{210}Pb supply results in an increase of sedimentation rate. This model could be useful in lakes and seas where sedimentation rates have been relatively constant through the study period. According to this mode, the specific activity $A(z)$ of unsupported ^{210}Pb decreases exponentially with the depth (Appleby and Oldfield, 1983).

$$A(z) = A_{max} e^{-\frac{\lambda z}{r}} \quad (2.1)$$

where: A_{max} is the maximum specific activity of unsupported ^{210}Pb corresponding to the upper part of sediments; λ is the radioactive constant of ^{210}Pb , z represents the depth measured from the sediment surface; r is the sedimentation rate.

This model fails if the activity-depth curves deviate from exponential form.

The other model, known as Constant Rate of Supply (CRS) or Constant Flux (CF) model, supposes that there is a constant fallout of ^{210}Pb from the atmosphere to the water that determines a constant rate of supply of ^{210}Pb to the sediment (Goldberg, 1963; Appleby and Oldfield, 1978). Because the CRS model assumes constant fallout of ^{210}Pb , it also considers that no post-depositional mixing occurs. The CRS model is usually appropriate in water bodies where there has been little or no sediment disturbance. In the CRS model, the sediment accumulation rate can vary, resulting in changes in the initial unsupported amount of ^{210}Pb . Referred to also as the Constant Flux model, it is used to determine the age of a given depth from a ^{210}Pb vertical profile within a sediment column (Krishnaswami *et al.*, 1978).

Within this model, the cumulated unsupported ^{210}Pb inventory decreases exponentially with depth, regardless of the variability of sedimentation rate, so that the ages t of older sediments are calculated not from their present concentrations but from the distribution of ^{210}Pb in the sediment record according to equation (Appleby and Oldfield, 1992; Sikorski and Bluszcz, 2003):

$$t = \frac{1}{\lambda} \ln \frac{C_C(0)}{C_C(z)} \quad (2.2)$$

where: $C_C(0)$ is the total surface activity of the unsupported ^{210}Pb in sediment;

$$C_C(z) = \int_0^z c_C(z) dz$$

is the total cumulate specific activity of unsupported ^{210}Pb considered from surface to depth z ; $c_C(z)$ is the specific surface activity of unsupported ^{210}Pb at depth z .

The CIC models has been applied successfully in large lakes or seas while the CRS models works best in medium-sized or small lakes (Binford, 1993).

^{137}Cs method

While ^{210}Pb is generated by the decay of radioactive ^{222}Rn and thus, its atmospheric content is constant over long periods of time, anthropogenic ^{137}Cs was released into atmosphere episodically between 1953 and 1963 as a result of nuclear atmospheric tests and in 1986 following the Chernobyl catastrophe. Absorbed irreversibly onto

sediments by clay, ^{137}Cs acts rather as a marker of the 1963 maximum and 1986 Chernobyl event (Geyh and Schleicher, 1990).

Although ^{137}Cs can diffuse through sediment, in the absence of bioturbation, the nuclear tests as well as the Chernobyl maxima preserve their positions in the sediments and thus can be used to ascertain a correct age to the observed maximum. The only relative disadvantage is related to the thickness of sediment slice used as the uncertainty in attributing to a given horizon the correct age is of the order of magnitude of the slice thickness.

As ^{137}Cs and ^{210}Pb methods are totally independent, but cover almost the same interval of time, the best results are obtained when these methods are used in tandem (Geyh and Schleicher, 1990; Appleby, 2000).

3. RESULTS AND DISCUSSION

Fig. 1a illustrates the vertical profile of the total as well as supported ^{210}Pb along the first 15 cm of the sediment. At the same time, the specific activity of supported ^{210}Pb as determined by measuring ^{226}Ra activity along the same segment of the core varied between 21.9 and 60.4 Bq/kg with an average value of 41.9 ± 8.5 Bq/kg. In these conditions, we have fitted, by using a nonlinear least square method, the vertical profile of ^{210}Pb specific activity $A(z)_{\text{total}}$ with a sum between an exponential corresponding to unsupported ^{210}Pb as described by Eq. 2.1 and a constant representing the supported ^{210}Pb specific activity:

$$A(z)_{\text{total}} = A_{\text{sup}} + A_{\text{unsp,max}} e^{-\lambda \frac{z}{r}} \quad (3.1)$$

where: A_{sup} represents the specific activity of supported ^{210}Pb ; $A_{\text{unsp,max}}$ is the maximum activity of unsupported ^{210}Pb which corresponds to the upper part of sediments; λ is the radioactive constant of ^{210}Pb , z is the depth meas-

ured from the sediment surface; r represents the sedimentation rate.

As results of fit we have obtained the following values: $A_{\text{sup}} = 55 \pm 9$ Bq/kg, $A_{\text{unsp,max}} = 912 \pm 44$ Bq/kg, $r = 0.49 \pm 0.03$ mm/y.

We consider the CIC model as the most appropriate because the relatively great depth of the collected sediment (about 600 m water depth) justifies the assumption of no mixing of the surface sediment. Another reason for using the CIC model is the relatively long residence time of the sea water, long enough to allow all available ^{210}Pb in the water column to be deposited on the sediment bed (Appleby *et al.*, 1979; Binford *et al.*, 1993; Blais *et al.*, 1995). Finally, the almost exponential form of ^{210}Pb profile as stated by Appleby and Oldfield (1983) was the third argument in the favor of CIC model.

For calculating the ^{137}Cs sedimentation rate we use the 1986 Chernobyl fallout depth. The maximum concentration of ^{137}Cs recorded by both laboratories was in the 0.5-1 cm layer. Knowing that the age of supply is 18 years (the sample was collected in 2004), the calculated sedimentation rate was 0.42 ± 0.20 mmy^{-1} (Fig. 1b).

Regarding ^{137}Cs and ^{210}Pb chronology, within the CIC model, the lead method is more suitable as it is based on a large numbers of determinations while radiocaesium dating based only on one single point. In our case, the maximum concentration of ^{137}Cs was registered in the second layer of the sediments, i.e. between 0.5 and 1 cm depth, which introduces a significant uncertainty in calculating the sedimentation rate.

Indeed, the sedimentation rate derived from the application of the ^{210}Pb CIC dating model was 0.49 ± 0.03 mmy^{-1} while the sedimentation rate, calculated by taking into account the position of ^{137}Cs specific activity maximum, varies between 0.27 and 0.55 mmy^{-1} (average 0.42 mmy^{-1}). These results can be considered satisfactory regarding the errors concerning the ^{137}Cs measurements,

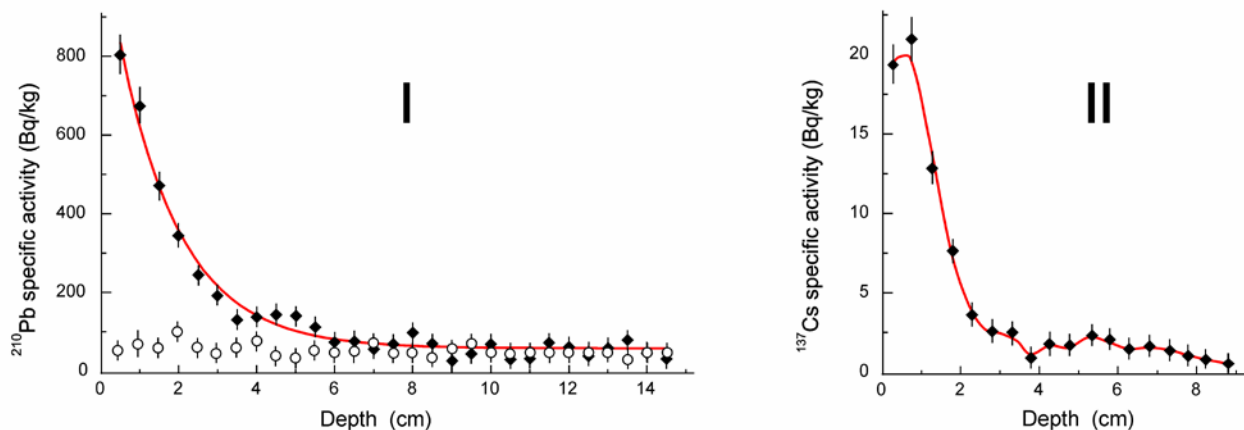


Fig. 1. Vertical profile of total (black circles) and supported (hollow circles) ^{210}Pb specific activity together with the corresponding best fit exponential decay curve according to Eq. 3.1 (continuous line) (I) as well as the vertical profile of ^{137}Cs (II). In all cases the specific activity was expressed in Bq/kg.

which are quite large relatively to ^{210}Pb dates errors (for ^{210}Pb sedimentation rate the errors are of about 5%).

As a result and within experimental uncertainties mainly associated with the lack of precision concerning the exact depth of ^{137}Cs maximum, the ages calculated by these two methods can be considered as coincident, enabling us to consider the ^{210}Pb sedimentation rate as the most suitable to calculate the age of the entire core. So, by considering the accumulation process as stationary for the time interval corresponding to the entire core, as radiographic images suggest, we have estimated the age of the entire 50 cm core to be roughly of one thousand years.

4. CONCLUDING REMARKS

Vertical profiles of both natural ^{210}Pb and anthropogenic ^{137}Cs were radiometrically determined by means of high resolution gamma ray spectrometry in a core consisting of unconsolidated sediments collected from the anoxic zone of the Black Sea at a water depth of 600 m below sea surface. Sedimentation rate determined within a Constant Initial Concentration model for ^{210}Pb accumulation gave a value of $0.49 \pm 0.03 \text{ mmy}^{-1}$, coincident within experimental uncertainties with that determined using Chernobyl ^{137}Cs maximum layer and equal to $0.42 \pm 0.20 \text{ mmy}^{-1}$.

The discrepancy in the total uncertainties between the two methods is related to the finite thickness of measured layers and to their number. Hence, while in the case of ^{137}Cs its maximum activity was attributed to a single layer, in the case of ^{210}Pb , its distribution was along more layers, which allowed a significantly better fit.

By taking into account the almost exponential profile of ^{210}Pb , together with the lack of bioturbation characteristic for the anoxic basin of the Black Sea, as well as the radiographic images of the core which shows an alternation of almost constant thickness laminae, we could suppose that at least the uppermost 50 cm of sediment was accumulated with an almost constant sedimentation rate, which finally gave an age of one thousand years for the oldest sediments of this core.

ACKNOWLEDGEMENT

This work was done within University of Bucharest Doctoral School (Grant 16513/FIZICA/56) and Ministry of Education and Research Grant PNII 31-068/2007 – CLIMARISC. We would also wish to thank to two anonymous referees for their useful remarks and suggestions.

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