Variations in ^{14}C ages of various organic fractions in a turbidite sediment core from Suruga Trough

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Radiocarbon (^{14}C) ages have been measured for four organic-carbon fractions, i.e., humic acid and solid organic materials of plant residues, and humic acid and humin of sandy-mud component, separated from a turbidite sediment, with a Tandetron accelerator mass spectrometer at Radioisotope Center, Nagoya University. The KT7819–24 piston-cored sediment, 275 cm in length, was collected at Suruga Trough, off Suruga Bay. The sediment materials from the lowermost turbidite bed of the cored sediment were used for the ^{14}C analysis.

Different organic fractions from the turbidite sediment showed relevant ^{14}C ages. The non-hydrolyzable humic acid of the plant materials was the youngest, with a ^{14}C age of 270 ± 80 y BP. The solid substances of the plant fragments showed a much older age of 1550 ± 130 y BP. The non-hydrolyzable humic acid from the sandy mud was dated to be 2270 ± 90 y BP, and the humin component was to be the oldest, with a ^{14}C age of 9400 ± 170 y BP. Concerning the chemical and physical character of the sedimentary organics in the turbidites, and their adsorption and mixing mechanism, it is inferred that the turbidite bed was produced shortly after 1550 ± 130 y BP, which is the age of the solid substances of the plant residues.

INTRODUCTION

Marine sediments are composed mainly of sand and clay particles from terrigenous rocks, volcanic substances, biological residues, and inorganic chemical substances deposited from sea water. In the bathyal and some abyssal regions near the lands, terrigenous mterials are the main constituents of the bottom sediments. The importance of turbidity currents, which transport the terrigenous sediments to the deep sea floor, has been well recognized. Clastics transported from land to sea by the river water flow deposited temporarily in the coastal regions on continental shelves and in canyon heads. These soft unsolidified sediments, when over-burdened, tend to slump suddenly, especially when triggered by earthquakes, tsunamis, and erruptions of oceanic volcanoes, and mix with sea water to produce mobile suspended matter with a density greater than that of the surrounding clear water. The suspended matter, when flowing swiftly down the continental slopes, forms a turbid density-current (a turbidity current), by stirring up sediments and by scraping and corroding materials on continental slopes. Thus, a large amount of terrigeneous materials can be transported to the abyssal regions mainly by the turbidity currents. It should also be recognized that overburdened sediments can slump down even without a special trigger when they build up at the head of an exceptionally steep slope of a deep bay or a trough such as that of the Suruga Bay, off the mouth of Fuji River, Central Japan. Such a slump and a turbidity flow actually took place on 12th of July, 1972 at Sagami Trough, which is on the opposite side of the Izu Peninsula from the Suruga Trough (Ōtsuka *et al.*, 1973).

In the bathyal and some abyssal regions, the

entire sedimentary sequence generally presents a rhythmic succession of coarse- and fine-grained beds, by a combination of the sediments deposited from turbidity currents, turbidites, and the hemi-pelagic mud deposited there steadily. A turbidite bed is characterized by grain-size grading, specific laminations, and special marks on the sole of the bed. Since turbidity currents occur intermittently, the dominant or main portion of the sediment in the deep region, where such turbidity curents can reach frequently, is com-

monly composed of sets of turbidites. Some turbidites may have correspondence with relevant trigger events, such as floods, earthquakes, or erruptions of volcanoes, recorded in history (Seibold and Berger, 1982).

Notwithstanding the important role of the turbidity currents in sediment accumuration on deep seafloor, any detailed discussion on the significance of ^{14}C age measurements of the turbidites has apparently not been published to date, except a preliminary work by Shiki *et al.*
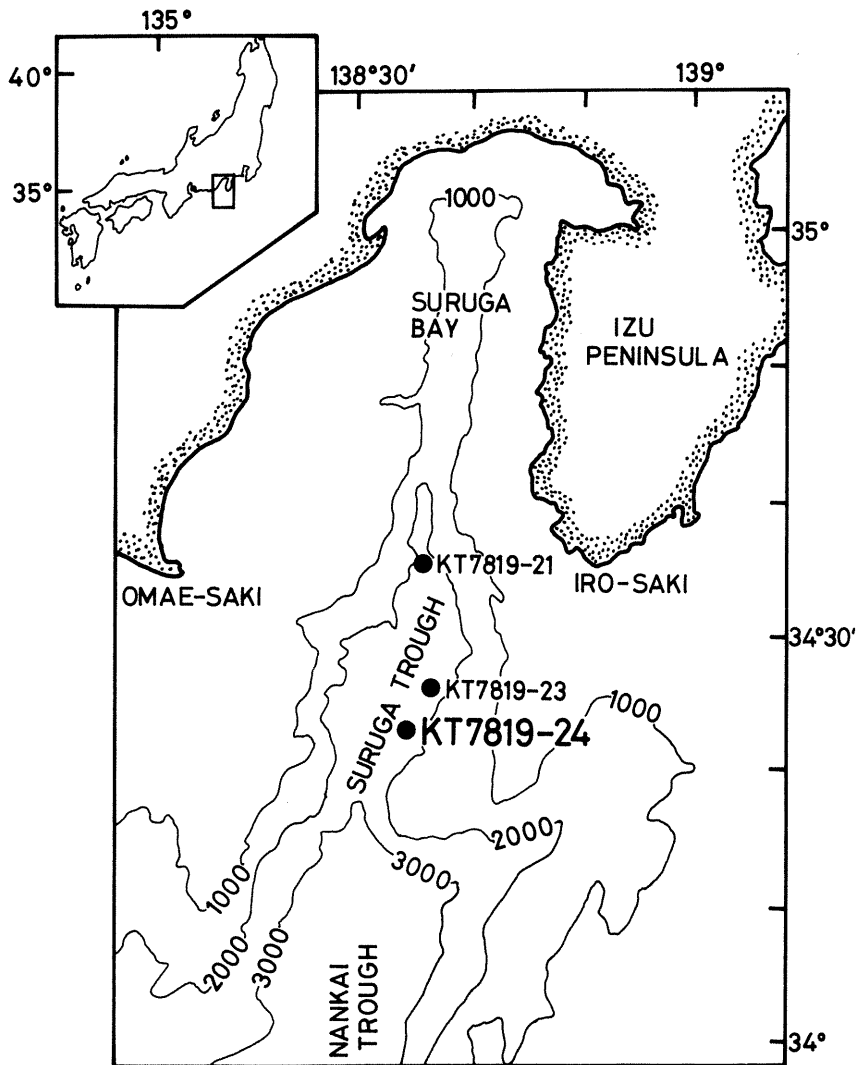

Fig. 1.   *Location map of piston-cored sediments. Code numbers of the cored sediments and contour lines with water depth in meter are given. KT7819–24 was analyzed for the present study.*
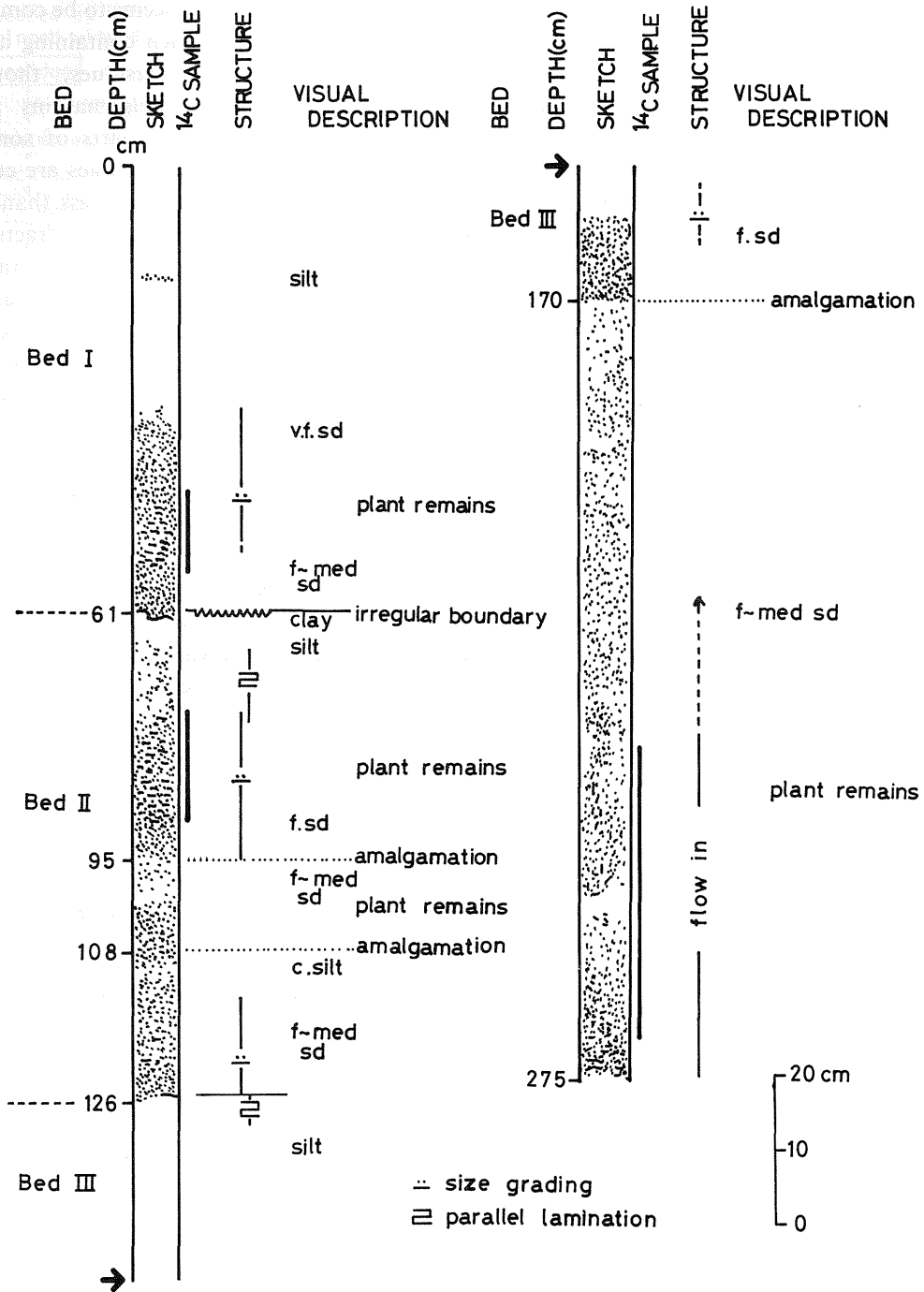
Fig. 2. Stratigraphic description of the KT7819-24 cored sediment. v.f.: very fine, f: fine, med: medium, c: coarse, sd:sand. Radiocarbon measurements have been carried out by Shiki et al. (1989) for the bulk samples collected from the three turbidite beds (45–56, 75–90, 220–270 cm deep from the sediment surface), and by the present study for the organic fractions separated from the lowest part of the same core (220–270 cm deep).

(1989).

Suruga Trough (2000 to 3000 m in depth), composing a long narrow concave area in the south of Suruga Bay, is a pathway of turbidity currents down to the Nankai Trough (3000 to 4800 m in depth), which proceeds southerly from Suruga Trough. Three piston cores were obtained in 1978, from the Suruga Trough as shown in Fig. 1, during the KT78–19 cruise of Tansei-maru, Tokyo University. One of the piston-cored sediments, 275 cm in length (KT7819–24), obtained at the 2890 meter-deep axis of the Suruga Trough, showed the characteristic structure of turbidites such as grading, laminations, and sole markings, stated above (Fig. 2).

Shiki *et al.* (1989) have obtained three ^{14}C ages for bulk sediment samples including plant residues and sandy mud at three horizons (Fig. 2), each corresponding to an independent turbidite bed of the cored-sediment (KT7819–24), by using a liquid scintillation method (Yamada and Kobashigawa, 1986). The resulting age-depth relation was rather contradictory; the deepest was the youngest (920±80 y BP, KSU-803), the uppermost was the second (1150±70 y BP, KSU-1521), and the central was the oldest (1919±90 y BP, KSU-1522). The plant residues separated from the deepest sediment sample were also dated. The ^{14}C age for the plant residues (550±70 y BP, KSU-1780) was far younger than that for the bulk sediment. To explain the results by Shiki *et al.* (1989), sources of organic materials in the turbidites should be clarified. In the present study, therefore, ^{14}C ages were measured for four organic fractions separated from the deepest turbidite that was analyzed by Shiki *et al.* (1989), with a Tandetron accelerator mass spectrometer at Radioisotope Center, Nagoya University (Nakai *et al.*, 1984; Nakamura *et al.*, 1985; 1987). The ^{14}C ages of the components are discussed in connection with the sources of those organic components in the turbidite.

SAMPLES

The cored sediment seems to be composed of three turbidite beds, each containing a portion with abundant plant residues, though the presence of a few amalgamatoins indicates reworking of the muddy parts of some other beds (Fig. 2). The plant residues are composed of very thin plant fragments less than 1 cm in length, produced probably by fracturing of leaves, barks, and twigs during the transportation with each turbidity current. The sandy mud and plant residues of each turbidite look rather new, showing no evidence of diagenesis.

A carbon-isotopic analysis was performed for sediment materials from the portion relatively abundant in plant residues, of the third turbidite from the sediment surface (220–270 cm deep), i.e., the lowest one observed in the KT7819–24 cored sediment (Fig. 2). Because of flow-in disturbance structure observed in the sampling portion of the core, further fine separation of the portion was not intended. The plant residues were separated by hand-picking from the sediment. Measurements of ^{14}C age and ^{13}C/^{12}C ratio were carried out for four organic fractions, namely, a NaOH-solution extract (humic acid) and a residual portion (solid carbon remains or humin), chemically fractionated from the plant residues and the resulting sandy-mud component of the turbidite.

EXPERIMENTAL PROCEDURES

Sample Preparation

The sediment sample was assayed for ^{14}C/^{13}C and ^{13}C/^{12}C ratios in the following manner. Plant residues such as small fragmental twigs, leaves, and roots were picked up by hand from the sediment sample of 10.6 g. Out of the resulting clastic component, 8.6 g was separated and distilled water was added. The mixture was stirred well and then decanted to eliminate fine plant fragments floating over the water.

Procedures of separating humic-acid and solid organic fractions for the plant residues and for the sandy-mud component are shown in Fig.

```
sample  (sandy-mud, plant residues)
   │←─1N HCl, 100ml
heated at 90°C for 1 hr.,
rinsed with distilled water
   │
 residue
   │←─0.5N NaOH, 100ml
heated at 90°C for 3 hr.
   │
decantation ─────────────────────→ solution 1 ──→ filtration through glass fiber
   │                                                   │
 residue                                            solution
   │←─0.5N NaOH, 100ml                                 │←─Conc. HCl(make pH<1.0)
heated at 90°C for 3 hr.                               │
   │                                                   │
decantation ─────────────────────→ solution 2 ──→ centrifugal separation
   │                                                   │
 residue                                            residue
   │←─0.5N NaOH, 100ml                                 │←─0.5N NaOH, 5ml
heated at 90°C for 3 hr.                               │
   │                                                   │
decantation ─────────────────────→ solution 3 ──→ centrifugal separation
   │                                                   │
 residue                                            solution
   │←─0.5N NaOH, 100ml                                 │←─Conc. HCl(make pH<1.0)
heated at 90°C for 3 hr.                               │
   │                                                   │
decantation ─────────────────────→ solution 4     centrifugal separation
   │                                    │              │
 residue                             abandoned      residue
rinsed with distilled water             │              │←─6N HCl, 50ml
   │                                    │           heated at 90°C for 3 hr.
 residue                             mingled        centrifugal separation
   │←─1.2N HCl, 200ml                   │              │
heated at 90°C for 3 hr.,               │           residue
decanted                                │              │
   │                              treated as       rinsed with distilled water,
 residue                          solution 1       dried at 100°C
   │←─1.2N HCl, 200ml                   │              │
heated at 90°C for 3 hr.,               │              │
decanted,                               │              │
rinsed with distilled water             │              │
   │                                    │              │
 residue                                │              │
   │                                    │              │
dried at 100°C                          │              │
   │                                    │              │
 humin component               humic acid 2 ──→ humic acid 1
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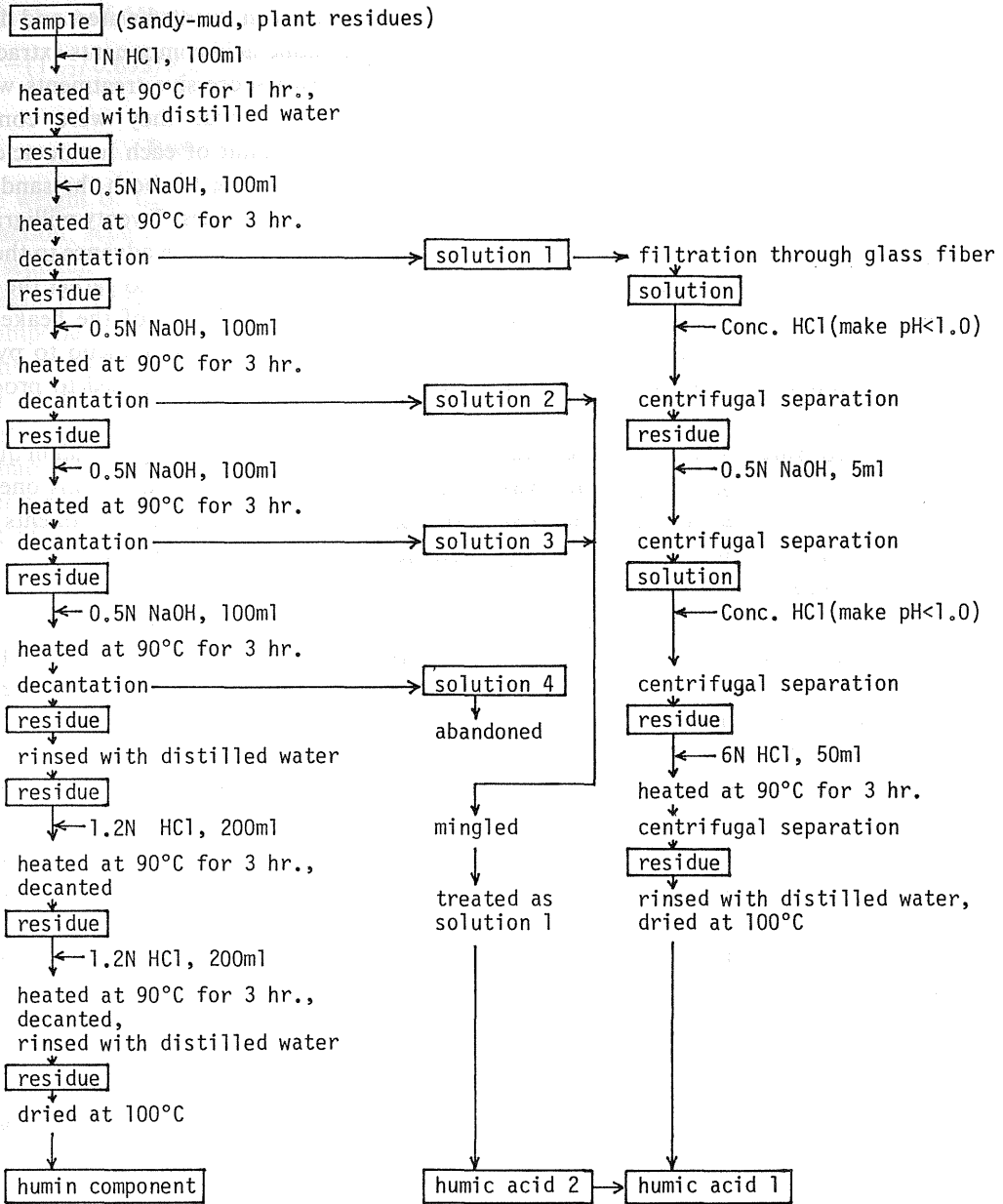
Fig. 3. Chemical procedures of separating humic-acid and solid organic fractions from plant residues and sandy-mud component. The humic-acid 2 was added to the humic-acid 1 before the pyrolysis for the carbon isotopic assay.

3. Both the plant materials and the clastics were treated with 100 ml of 1 N HCl for an hour at 90°C to eliminate carbonate and fluvic acid. The two samples, rinsed with distilled water, were mixed with 0.5 N NaOH of 100 ml and heated at 90°C for 3 hours, to extract base-soluble materials. The solution was separated from the solid residue by decantation. This extraction procedure was repeated four times. Since the third extract appeared to be so small in quantity, indicated by the thin brown color of the solution, it was added to the second. The fourth extract,

which appeared to be far smaller in quantity than the third, was abandoned.

The resulting solid residues, after extracting humic acid from both samples, were rinsed with distilled water and then treated with 1.2 N HCl for 3 hours. The acid treatment was performed two times. The solid samples were rinsed again with distilled water, and dried at 100°C.

The solid materials from plant residues were sealed in a pyrex ampoule under vacuum and heated at 450°C for 2 hours. The pyrolyzed sample was again treated with 1.2 N HCl at 90°C for 2 hours to eliminate any residual carbonates, rinsed with distilled water, and dried. The sample thus pyrolyzed, weighing 5.0 mg in all, was mixed with 32.6 mg of silver powder. The mixture was compressed, by using a hand compressing tool, to produce a carbon-silver mixture target for the $^{14}C/^{13}C$ ratio measurement with the Tandetron spectrometer.

The humin fraction of the sandy-mud component was extracted in the following manner. The sandy-mud sample of 1.95 g, after the humic-acid extraction, was mixed with 2.52 g of cobalt oxide (Co_2O_3) and heated in vacuo at 950°C to produce CO_2, in total 5.0 mg in carbon (0.26% in weight ratio to the sandy-mud), which was then reduced to elemental carbon by metal magnesium (88.7 mg) at 950°C. After treatment for an hour with 6 N HCl, the amorphous carbon was filtered through teflon and dried. The amorphous carbon, weighing 3.6 mg in all, was mixed with 43 mg of silver powder to produce a C–Ag target.

Two samples of base-soluble materials were filtered through glass fiber (GB-100R, heated at 500°C for 2 hours in advance) to eliminate any solid substances. Humic acid of each sample was precipitated by adding concentrated HCl to the solution to make its pH value lower than 1. The deposits were separated and rinsed with distilled water in a centrifuge. The humic-acid deposit was purified by re-dissolving with small amount of 0.5 N NaOH, reprecipitating with concentrated HCl, and separating with a centrifuge. The humic acid was then treated with 6 N HCl for 3 hours at 90°C to eliminate the hydrolyzable

fraction, rinsed with distilled water, and dried at 100°C. The humic-acid components extracted by the first and two successive treatments were so small in quantity that they were combined together. The amount of each humic-acid sample was less than 1 mg for both the sandy-mud and plant-residue samples. Twenty milligrams of silver powder were added in advance to the dried humic acid to make it easier to gather the humic acid spread over the inside of the beaker. The mixture, heated at 450°C in vacuo to pyrolyze the humic acid, was then pressed to produce a C–Ag target.

The prepared carbon was so small in quantity that all of it was used to produce only one target for each of the four organic components of the sediments.

Carbon Isotope-ratio Measurement

First, the $^{14}C/^{13}C$ ratio, $\delta^{14}C(13)$, was measured in the Tandetron spectrometer, and given as follows:

$$\delta^{14}C(13) = \left(\frac{(^{14}C/^{13}C)_{sample}}{(^{14}C/^{13}C)_{standard}} - 1 \right) \times 1000 \quad (‰). \quad (1)$$

As a radiocarbon standard, charcoal prepared from 1840–1860 annual rings of a Japanese cypress tree which had $^{14}C/^{13}C$ ratio calibrated to NBS oxalic acid (SRM-4990) was used (Nakamura *et al.*, 1985). Next, the sample $^{13}C/^{12}C$ ratio normalized to that of the PDB-Chicago standard, $\delta^{13}C_{PDB}$, defined as follows,

$$\delta^{13}C_{PDB} = \left(\frac{(^{13}C/^{12}C)_{sample}}{(^{13}C/^{12}C)_{PDB}} - 1 \right) \times 1000 \quad (‰), \quad (2)$$

was measured with a MAT-250 mass spectrometer at the Water Research Institute, Nagoya University, for CO_2 produced by combusting (Minagawa *et al.*, 1984; Kitagawa, 1987) the remaining C–Ag target which was used for the ^{14}C measurement. The sample $\Delta^{14}C$ value, corrected for isotopic fractionation, was then calculated as

$$\Delta^{14}C = \delta^{14}C(13) - (25 + \delta^{13}C_{PDB})$$
$$\times (1 + \delta^{14}C(13)/1000) \quad (‰). \quad (3)$$

The ^{14}C age (T) was calculated by using the ^{14}C

half life ($T_{1/2}$) of 5570 years as follows:

$$T = -(T_{1/2}/0.693) \times \ln (1 + \Delta^{14}C/1000). \quad (4)$$

RESULTS

The weight of the non-hydrolyzable humic-acid fraction, separated from the plant residues in the sediment of 10.6 g, was less than 1 mg. The weight of the same fraction from the sandy-mud component of 8.6 g was also less than 1 mg. According to Nakamura *et al.* (1989), the carbon content of the non-hydrolyzable humic acid, prepared by the procedure described in the text from charcoal collected at an archeological site, has been measured to be about 56%. The carbon content of the sandy-mud component after extracting humic-acid was found to be 0.26%. Thus the major organic-carbon fraction of the turbidite analyzed here was the humin of the sandy-mud component, as summarized in Table 1.

The values of δ^{13}C, ranging from -25.6 to -27.0‰ for four organic fractions of the sediment, suggest that these fractions have been produced prominently from terrestrial C3 plant materials (Wada, 1986).

A distinct discrepancy of ages for different fractions was found, i.e., the ^{14}C ages varied from 270 to 9400 y BP. The non-hydrolyzable humic acid of the plant residues was the youngest, showing the ^{14}C age of 270 ± 80 y BP (NUTA-749). The solid substances of the plant residues showed a much older age of 1550 ± 130 y BP (NUTA-715). The ^{14}C age of the non-hydrolyzable humic acid from the sandy mud, 2270 ± 90 y BP (NUTA-748), was older than that of each fraction from the plant residues. The humin component from the sandy-mud was the oldest, showing the ^{14}C age of 9400 ± 170 y BP (NUTA-750).

Table 1. *Values of carbon content, $\delta^{13}C_{PDB}$, $\Delta^{14}C$, and ^{14}C age for four organic-carbon fractions from the third turbidite of the KT7819–24 piston-cored sediment*

Sample	Measured fraction	carbon[a] content (%)	$\delta^{13}C_{PDB}$ (‰)	$\Delta^{14}C$ (‰)	^{14}C age (y BP)	Measurement code
plant residues	humic acid, treated with 6N HCl at 90°C	<0.0053	−25.6	−33.2±9.4	270±80[b]	NUTA-749
plant residues	solid substances after acid and base treatment	0.047	−27.0	−175.7±13.1	1550±130[b]	NUTA-715
sandy-mud	humic acid, treated with 6N HCl at 90°C	<0.0065	−26.7	−246.4±8.6	2270±90[b]	NUTA-748
sandy-mud	humin	0.26	−26.0	−689.3±6.7	9400±170[b]	NUTA-750
plant residues	total				550±70[c]	KSU-1780
mixture of plant residues and sandy mud	total				920±80[c]	KSU-803

[a] *Carbon content of each organic fraction is given in the weight percent to the original sediment sample. The carbon content of solid substances from the plant residues were calculated by assuming that the solid substances after pyrolysis consist of carbon only.*

[b] *Based on the ^{14}C half life of 5570 years; 0 y BP=A.D. 1950, and corrected for carbon isotopic fractionation.*

[c] *Based on the ^{14}C half life of 5568 years (Shiki et al., 1989).*

DISCUSSION

Age Discrepancy among Four Organic Fractions

Since humic acid in the sediments can be transported vertically by pore water (Kigoshi *et al.*, 1978), humic-acid fractions of plant residues and sandy-mud component at any horizon of the sediments may partly contain foreign humic acid transported from upper and lower horizons. On the contrary, solid organic fractions in the sediments, insoluble in water, as well as acid- and base-insoluble, are not movable and are preserved in the sediments where they accumulated, if there is no turbulent movement of the sediments after they deposited. Therefore, the ^{14}C ages of solid materials from the plant residues and of humin from the sandy-mud component should be discussed first.

Fragments of wood and grass are rather abundant in the turbidites (*ca.* 15% of total organic carbon in the turbidite studied here as shown in Table 1), compared with hemi-pelagic muds accumulated continuously. These plant fragments must have been carried from land to sea through rivers mainly during floods of a large scale, induced by heavy rains and storms (Otsuka *et al.*, 1973). The plants could have been deposited only very temporarily on the continental shelves, and were transported by a turbidity current, accompanied with sand and clay particles, to deeper sea regions of Suruga Trough at water depths of over 2000 m. Thus the major part of the plant residues, accumulating abundantly in the turbidite sediment, may have been introduced by such an event.

The ^{14}C age of humin separated from the sandy-mud component of the turbidite is far older than that of the solid plant materials, which were handpicked out of the same turbidite. This fact indicates that such humin originates not only in smaller fragments of plant residues, incapable of being picked up by hand, but also prominently in older organic matters (plankton produced in sea water, fine organic particles derived from land, etc.) which had originally accumulated simultaneously with sand and mud particles to form temporal sediments, before those sediments were involved in the turbidity current. Thus these organic matters can be supplied from older sediments which had deposited steadily and successively and stayed rather long on the continental shelves. Slumping of such sediments, when overburdened by a large-scale flood, may produce a turbidity current, at the head of the sub-marine canyons which develop at the shoulder of very narrow continental shelf of the Suruga Bay. In addition, dead carbon, containing no ^{14}C, in organic compounds of the sedimentary rocks, from which sand and mud particles were produced, may also contribute to the old ^{14}C age of the humin fraction (Nakamura *et al.*, 1987).

The humic acid extracted from the plant residues shows a younger age than the solid fraction of the residues. This indicates that a part of the humic acid, younger than the solid fraction of the plant residues, may have been transported from the organic materials in other horizons, perhaps in the upper horizons, through pore water in the sediments. The humic acid was then adsorbed on those plant residues to be mixed with the humic acid of the older age which had been produced authochtonously from the plant residues. The humic acid extracted from the sandy-mud component of the sediment also shows a younger age than the humin from the same component. This can also be explained qualitatively, partly by the movement of humic acid, i.e., humic acid which is transported from upper horizons through pore water to be exchanged, and partly by the addition of the humic acid which was produced authochtonously by the decomposition of the plant residues.

Comparison with Other Experimental Results

Shiki *et al.* (1989) have obtained two ^{14}C ages for the same turbidite analyzed in this study (Table 1). The age of 920 ± 80 y BP was obtained for the bulk sediment containing both sandy-mud and plant residues, and 550 ± 70 y BP for the plant residues selected by floating them over water. These ^{14}C ages were measured at Kyoto Sangyo University (Yamada and Kobashigawa, 1986) in the following manner. Each dried sam-

ple was heated in an airtight electric furnace at 800°C for 2 hours, and then placed in a quartz tube and subjected to a stream of heated N_2 gas for one hour at 500°C. Next, O_2 was passed through the tube to convert sample carbon into CO_2 completely. The CO_2 was then changed to methanol to measure [14]C activity by the liquid scintillation counting method. Thus no chemical pre-treatment, for separating fulvic and humic acids, was performed in the study by Shiki *et al.* (1989).

The result that the [14]C age was younger for the selected plant residues than for the bulk sediment was consistent with the present study. However, the [14]C ages by Shiki *et al.* (1989) were unexpectedly young. Apparent [14]C ages for the bulk samples were estimated by using the present results, i.e., the carbon content of each organic fraction, the Δ[14]C value of each fraction, and eq. (4). The upper limit value of the carbon content was used for each humic acid, i.e., 0.005% for the humic acid from the plant residues and 0.007% for that from the sandy mud. The calculated [14]C values, 7160 y BP for the bulk sediment and 1430 y BP for the total plant residues, are far older than the relevant [14]C ages of 920 and 550 y BP, respectively, obtained by Shiki *et al.* (1989). The apparent differences in [14]C ages between the two measurements have not yet been explained quantitatively. However, this discrepancy can be partly explained by the chemical pre-treatment of the samples, in the following manner. Fulvic acid, extractable with an acid-treatment, has been observed to give younger [14]C age than do other organic components for soil samples (Campbell *et al.*, 1967; Miyazaki, 1971; Matthews, 1984). In the present analysis, fulvic acid was removed by an acid pre-treatment. On the contrary, fulvic acid, which would give younger [14]C age than humic acid, was included in the study by Shiki *et al.* (1989). Nevertheless, the fulvic acid content may not be large enough to fill up the gaps in [14]C ages.

The reversal of the [14]C ages obtained by Shiki *et al.* (1989) for three successive turbidites may be explained by the different mixing rates of the plant residues and the humin component.

Age of Formation of the Turbidite

The above discussion suggests that the time when the event, i.e., the over-accumulation of sediments by a flood of a large scale and subsequent collapse of those sediments, occurred, which successively induced the submarine slump or the turbidity current and finally produced the turbidite studied in this article, may be closely related with the [14]C age of the solid fraction of the plant residues; the age of the event is almost equal to or shortly after 1550 ± 130 y BP.

When the bulk sediment is used for [14]C dating, the results may sometimes be contradictory, as in the case of Shiki *et al.* (1989) mentioned above. This does not necessarily mean, however, that [14]C dating is useless for the age measurements of the sediments. It is suggested from the present study that more accurate ages could be obtained for the sediments by selecting an appropriate organic fraction in the sediment, and by applying an appropriate correction to the measured age, when necessary. To confirm this conclusion, however, such basic studies must be carried out for many sediments from various depths and various places in the ocean.

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