AGE ESTIMATION OF THE PLIOCENE AND QUATERNARY DEPOSITS IN HOKKAIDO BY AN AID OF SOLUBLE COMPONENTS OF FOSSIL WOODS WITH ACETYLBROMIDE TREATMENT

by

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(with 2 tables and 1 figure)

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Abstract

Survival percentage of the fossil woods with acetylbromide treatment, collected from various horizons of the Pliocene and Pleistocene deposits in Hokkaido have been tested by the authors, which shows rather regular decrease with age of the fossil woods as has been already well established by ITIHARA and others in the region around Osaka city. This fact may be accordingly applied to the stratigraphical correlation to some extent. The results of our study on the survival percentage determined by the material of Hokkaido will be briefly presented below.

INTRODUCTION

Y. ITIHARA *et al.* (1966) and N. KAGEMORI and M. ITIHARA (1967) introduced so-called acetylbromide method to age estimation of the Pliocene and Quaternary deposits developed around Osaka city in southwest Honshu, Japan. Namely, the main constituents of wood, cellulose hemicellulose and lignin, are soluble in acetylbromide, while humus matter transformed through diagenesis from the original material is not attacked by the reagent above mentioned. Original constituents of woods and humus matter may eventually still remain in fossil woods in a various degree, and it is supposed that an amount of surviving original matter of woods may be depending on a condition of diagenesis under which fossil woods have been preserved in deposits. In other words, it may be expected that there is no regular relationship between an amount of soluble matter in acetylbromide within fossil woods and their geologic age. Nevertheless, ITIHARA *et al.* (1966) and KAGEMORI and ITIHARA (1967) succeeded to establish a regular relationship between them, relative age

of the woods being estimated by various geological informations. Namely, the older in the geologic formation, the lesser amount of soluble matter in the fossil wood by an acetylbromide treatment.

In another words, the amount of soluble matter in acetylbromide gradually decreases in fossil woods from the younger towards the older formation also in Hokkaido. Therefore this method may be proved to be a fairly good aid for stratigraphical correlation of the younger Tertiary and Quaternary deposits developed in Hokkaido, just like in the case around Osaka city.

SAMPLES

The fossil woods to be discussed here are shown in Table 1, with their sampling localities, their geologic horizons and estimated ages. Brief explanations of the samples are given below for each horizon, respectively.
Sample no. 1: A weathered branch of the dwarf pine tree was collected at about 1200 m in altitude in the active volcano Tokachi-dake. Estimated age may be less than 100 years old (Coll.; M. MINATO).
Sample no. 2: A fossil branch of *Larix Gmelini* collected at Nishinosato, immediately south of Sapporo city is 22,700 ± 1,000 ys. BP (Gak–2216) by C^{14} age determination (YANO and FUJITA, 1970) (Coll.; S. KUMANO).
Sample no. 3: The fossil wood found from the peat layer in the gravel deposits

Table 1 Fossil woods samples from the Pliocene and Pleistocene deposits in Hokkaido with survival percentage soluble in acetylbromide

Sample No.	Locality	Formation	Estimated age ($\times 10^3$ yrs.)	Survival percentage
1	Tokachi-dake, Central Hokkaido	———	Recent	98.0
2	Hiroshima-cho, Sapporo-gun, Ishikari	Larix of Takadai clay bed	22.7 ± 1.0	94.7
3	Kuttari, Kamikawa, Tokachi	———	180 ~ 70	78.5
4	Nayoro city, Kamikawa	Hacchashinai	600 ~ 700	65.2
5	Piribetsu, Ashoro-gun, Tokachi	*Larix* Ikeda	1,850~2,000	34.0
6	Ikeda-machi, Nakagawa-gun, Tokachi	Lower Ikeda	1,850~2,000	21.2
7	Ikeda-machi	Lower Ikeda	1,850~2,000	21.2
8	Ashoro, Nakagawa-gun, Tokachi	Tobushi coal bearing	4,500	7.7
9	Ashoro	Inaushi coal bearing	5,000	0.0
10	Ashoro	Inaushi coal bearing	5 000	0.0
11	Ashoro	Ihaushi coal bearing	5,000	0.0

may be presumed to be of the later Riss-Würm Interglacial age, accordingly about 10^5 years old (Coll.; M. MINATO).

Sample no. 4: The fossil woods were collected from the peat deposits found at the basal part of the Hacchashinai formation developed around Nayoro city, northern Hokkaido. This formation is chiefly composed of unconsolidated gravel, sand and less amount of clay, intercalating a thin layer of peat or peaty material, no less than 100 m in total thickness. Similar type gravel deposits are widely distributed elsewhere in the Wakkanai district in the northernmost part of Hokkaido for example, and also in the central part of the island around Asahikawa city and Biei town, in the latter districts rhyolitic pumice flow being observed to cover the gravel bed with unconformity. The rhyolitic tuff is now correlated to the boundary of Matuyama/Bruhnes epoch by its paleomagnetic polarity, and the fossil wood may be, therefore, correlated to the "Donau-Schotter" in a wide sense, being estimated to be 700 – 800 x 10^3 years old (Coll.; M. MINATO).

Sample nos. 5, 6, and 7: Fossil woods were collected from the lignite or peat layer, stratigraphically situated in the lower part of the Ikeda formation. The rhyolitic pumice flow found just below the peat or lignite layer shows normal paleomagnetic polarity, and the dacitic flow above the carbonaceous layer also shows normal polarity. While MINATO *et al.* (1971) has already established the horizon of Jaramillo event in the stratigraphical sequence of the lower Quaternary deposits in Hokkaido far above the upper and lower Ikeda formation. The rhyolitic and dacitic tuff intercalated in the lower part of the Ikeda formation above mentioned, may be accordingly regarded to occupy the uppermost horizon of the Pliocene being now assumed to represent the Olduvai event.

According to MINATO *et al.* (1971), the Hacchashinai (gravel) formation shows rather cold climate than at the present, while the lower Ikeda formation indicates warmer climate. The Okuenhoro basalt which occupies stratigraphically below the lower Ikeda formation shows a reversed polarity. Hence, the stratigraphical horizon of the samples may be regarded to be the uppermost Pliocene, viz. about 1.85 – 2.00 x 10^6 years old (Coll.; M. MINATO and K. MITANI).

Sample nos. 8, 9, 10 and 11: Fossil woods collected from the Inaushi lignite bearing formation may be correlated to the Lower Pliocene, because *Pecten takahashii*, a Japanese Lower Pliocene leading scallop, is found in the Rawan conglomerate under the lignite bearing formation. The underclay of the Inaushi lignite bearing formation shows normal palaeomagnetic polarity. It may be inferred to be correlatable to the lower Gilbert reversed epoch or a little earlier phase than it, with high probability, viz. 4.50 – 5.00 x 10^6 ys. old in rough

estimation.

METHODS

Specimens for analysis were thoroughly scrubbed with distilled water. The woods were cut off into small pieces with knife and then were dried in an oven at 105° C for an hour. The dried specimens were crushed with stainless mortar into powder less than 80 mesh. After being dried again at 105° C, they were kept in a desiccator. The powdered sample (0.300 gm) were refluxing with 7 ml of acetylbromide for 72 hours at 40° C. The resulting black solution was filtered with a Gooch crucible under vacuum and the residue was then washed with acetylbromide. After the residual acetylbromide was evaporated at 90° C, the residue was washed with 20 ml of ether, 0.5 M potassium thiosulfate solution and hot water successively. This procedure made the original main constituents soluble, and humus materials and contaminated minerals left, if there were. After being dried in an oven at 110° C for 2 hours and kept in a desiccator, the crucible with residue was weighed. The difference of the crucible after burning out the residue was the humus weight (H) in the sample. The acetylbromide-soluble percentage S (AcBr) was calculated by the following formula.

$$S\ (AcBr) = \frac{W - H}{W} \times 100$$

If there were much minerals left in the crucible after burning, the original weight (W) of the sample should be corrected. All of this procedure has been established in the fossil wood treatment developed by ITIHARA, and others (ITIHARA *et al.*, 1966; KAGEMORI and ITIHARA, 1967).

RESULTS AND DISCUSSION

Survival percentages of the fossil woods soluble with acetylbromide are shown in the right column of Table 1, and are shown graphically in Fig. 1 with the relationship to absolute age. This graph is very similar to those obtained by ITIHARA and others (Itihara *et al.*, 1966, KAGEMORI and ITIHARA, 1967). It must be concluded that the survival percentage regularly decreases with the lapse of time, and that this decreasing rate shown in Fig. 1 may be accordingly applicable to the age determination of the unknown fossil wood samples, although the samples analysed here are limited in number.

It has been stated that the cellulose, hemicellulose and lignin of the main composition in woods are hydrolysed and the resulting low molecular weight

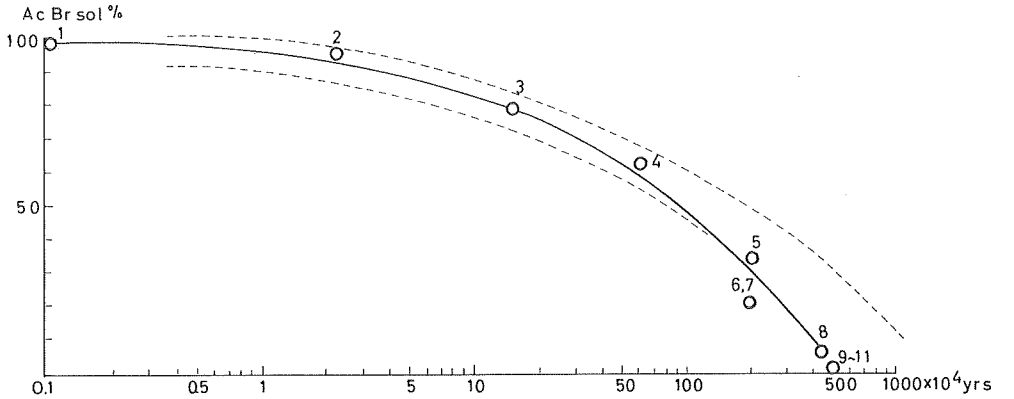

Fig. 1 Survival percentage of the fossil woods soluble in acetylbromide to the geologic ages. (Dotted lines are reproduced from KAGEMORI and ITIHARA (1967))

chemicals are then polymerized to humus insoluble with acetylbromide treatment. In natural conditions some parts of the hydrolysed materials dissolve away from the wood and the restricted parts are polymerized to humus. If this is the case, it is probable that the experiment mentioned in this paper can only detect the humus materials and the original amount of the wood component should be unknown. The production rate of humus in the course of diagenesis may be assumed to be independent on the hydrolysis rate, if there are enough amount of the hydrolysed matters to be polymerized. The humus production may be depending on the physico-chemical conditions of sediments in which woods were buried. Therefore, the humus content of the fossil woods decreases with the lapse of time without so much affection from the condition at different places.

To examine an availability of this age determination method, we selected two fossil wood samples from the upper peat bed and one sample from the lower peat bed, both in the Horokayanto formation, which typically develops in the low hills facing to the lake Horokayanto located at southeastern coast of the Tokachi province and chiefly composed of gravels and less amount of clay, sand and peat. There is an unconformity among the middle of this formation; the so-called upper peat belongs to the upper part of this formation, while the lower peat is the top member of the lower part of the Horokayanto formation (OSANAI *et al.*, 1971).

Now, the survival percentage of the wood collected from the upper peat is around 85% (Table 2), indicating about 70,000 ys., judging from the standard survival percentage curve obtained in this study (Fig. 1). This absolute age is almost corresponding to the earlier phase of the early Würm (Frühwürm) ice age (W 1).

Table 2 Analyses of the fossil woods from the Horokayanto formation, Hiro-o-gun, Tokachi, Hokkaido

Horizon in the Horokayanto formation	Acetylbromide-soluble percentage
Middle peat bed	85.4
	84.8
Lower peat bed	71.3

As a matter of fact, the upper peat layer of the Horokayanto formation indicates very colder climate than at present from the pollen assemblage (IGARASHI, Y. and KUMANO, S., 1971). Further, the Horokayanto formation is unconformably overlain by a thin loamy deposit including the Shikotsu pumice fall deposit dated as 32,000 ys. BP (C^{14} age). These facts above mentioned may well coincide with the estimated absolute age by acetylbromide method.

Further, the lower peat bed is now estimated as about 300,000 ys. BP. This estimated age may not be contradictory to the paleoclimate of the above-mentioned horizon, the top of the lower Horokayanto formation, which is also inferred from pollen analysis. It may be, in fact, correlatable to the late Mindel/Riss interglacial (MINATO and AKIYAMA, 1971).

Acknowledgement

We thank Dr. M. ITIHARA, Dr. Y. ITIHARA and Dr. N. KAGEMORI for giving us many suggestions and Dr. K. MITANI and Mr. S. KUMANO for offering some of the specimens treated in this paper. This research was supported by a grant of the Historical Museum of Hokkaido.

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(Manuscript received August 31, 1971)