Chemical characterization of peat by EGA-MS and multi-step Py-GC/MS methods

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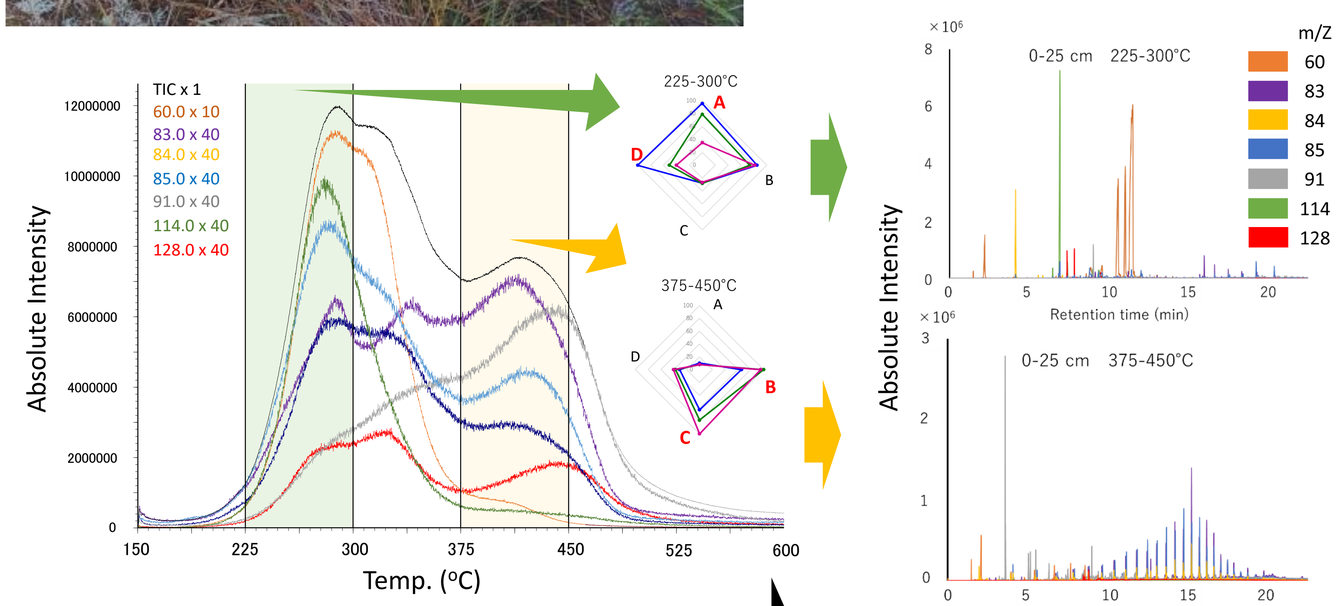
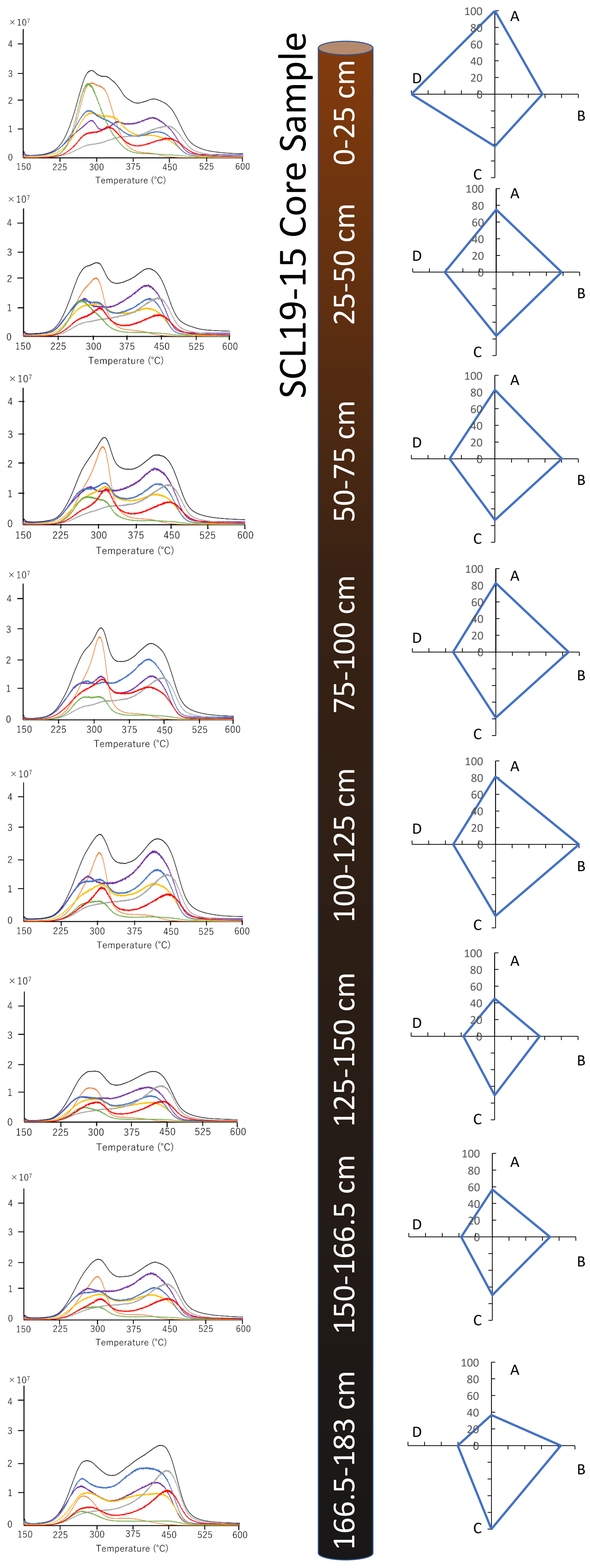
Sampling Sites

Sample name	Longitude	Latitude
SCL18		
1	57° 17’22.02”N	6° 21’56.13”W
3	57° 46’01.78”N	5° 44’59.34”W
9	57° 26’30.30”N	6° 45’12.08”W
10	58° 00’03.45”N	5° 08’00.77”W
13	58° 22’35.90”N	5° 03’17.81”W
SCL19		
15	58° 36’51.81”N	3° 05’00.78”W
17	58° 15’02.73”N	3° 32’22.98”W
21	58° 30’21.69”N	4° 32’37.27”W
24	58° 12’05.78”N	4° 30’58.55”W
26	57° 25’54.40”N	3° 39’31.36”W

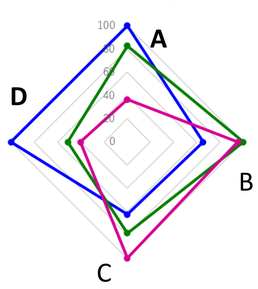


Thermogram of SCL19-15

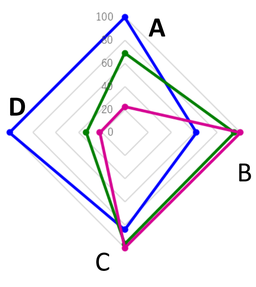
Pyrogram of SCL19-15

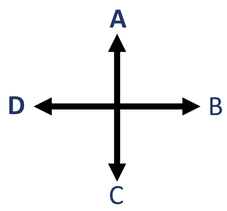


EGA-MS



ms-Py-GC/MS





0-25 cm
166.5-183 cm
75-100 cm

A: Derived from cellulose components
B: Derived from amino acid components
C: Derived from lignin components
D: Derived from aliphatic hydrocarbons

Thermogram &Component Evaluation

CONCLUSION

The gas emissions in each temperature fraction of the thermogram obtained simply by EGA-MS analysis can be analyzed by detailed analysis using the ms-Py-GC/MS method, and the results of the component evaluation based on each analysis method showed good agreement, indicating the validity of the simple evaluation method using EGA-MS. The similarity and uniqueness of the four components by region can be evaluated by EGA-MS analysis alone in the evaluation by location, and it is considered to be a useful analytical method.

INTROCUCTION

Peat is used to dry barley, the raw material for whisky, and has a great influence on the flavor of whisky. Therefore, chemical characterization methods that clearly show the characteristics of peat are necessary for whisky quality control. Many methods such as nuclear magnetic resonance have been attempted to evaluate peat. However, it has not been easy to classify them based on their chemical composition because their characteristics vary depending on environmental factors and time factors.

SAMPLINGS & EXPERIMENTS

Peat samples were collected at 12 sites in November 2018 (SCL18) and 13 sites in November 2019 (SCL19) for 30 cm core samples, and vertical samples were collected at 3 sites in November 2019.30 cm core samples were collected using a soil core sampler (hand sampler HS30S; Fujiwara Manufacturing; inner diameter 5 The 30 cm core samples were collected in a series of transparent PVC cylinders (for hand sampler; Fujiwara Mfg. Co., Ltd.) at each site using a soil core sampler (hand sampler HS30S; inner diameter 5 cm × length 30 cm) from 0 to 30 cm soil depth. Vertically deeper core samples were collected using a peat sampler (DIK-105A peat sampler for 5 m; Daikki Rika Kogyo; inner diameter 5 cm × length 50 cm, 0.42 L semi-cylindrical) in one series for 0-183 cm, two series for 0-147 cm, and five series for 0-100 cm at each site.

The EGA method involves a multi-shot pyrolyzer (EGA/PY-3030D; Frontier Labs) with an auto-shot sampler (AS-1020E; Frontier Labs) directly connected to a GC/MS (QP2010 Ultra; Shimadzu Corporation) with an inert capillary tube (D-EGA ; length 2.5 m, inner diameter 0.15 mmID). A 1.0 mg peat sample was weighed in an inert stainless cup (PY1-EC80F; Frontier Labs) and covered with quartz wool on top of the sample to prevent sample scattering. The sample cup was set in an auto-shot sampler and heated to 150-600 ° C at a heating rate of 15 ° C/min under a helium flow rate of 18.3 mL/min.

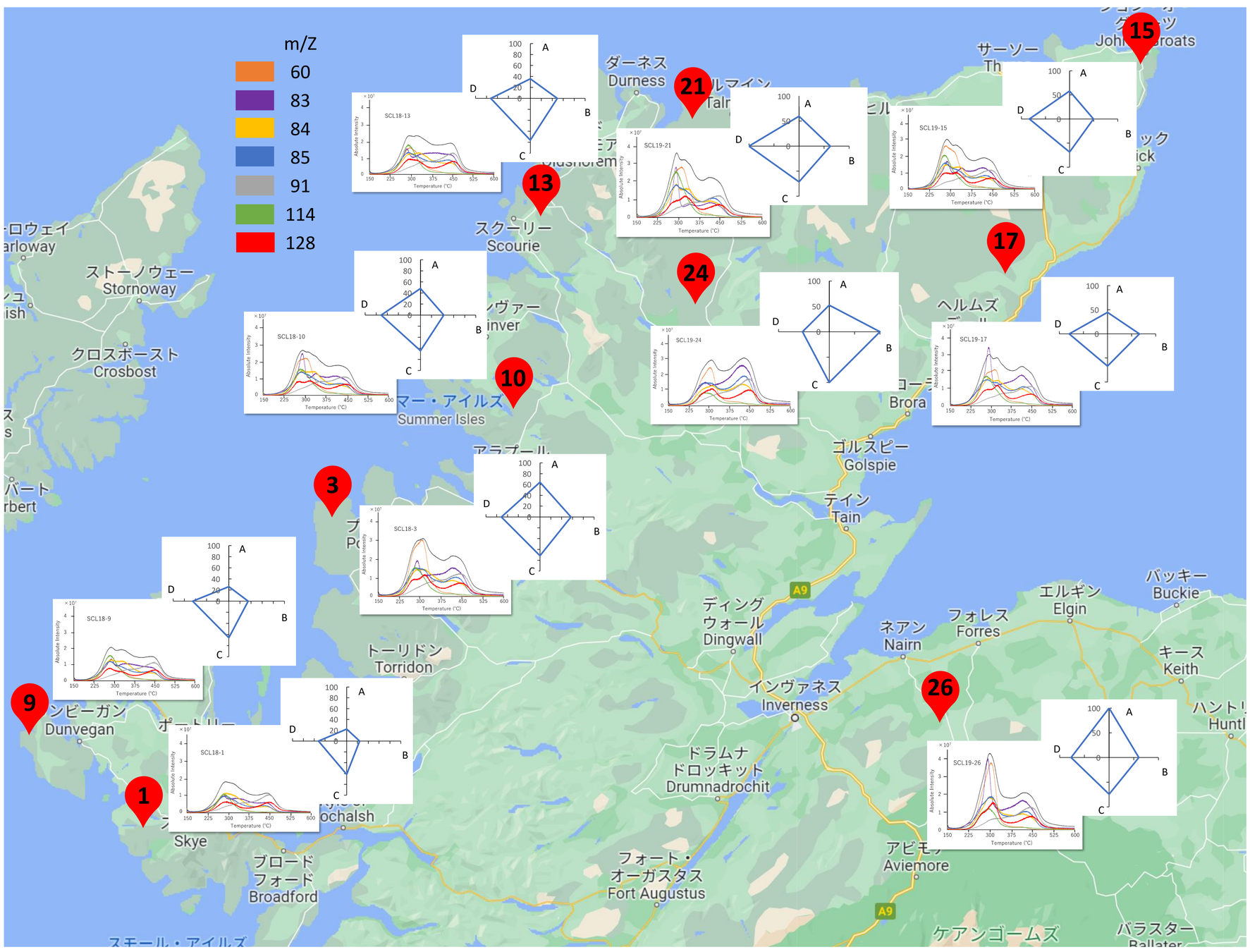
The multi-step(ms)-Py-GC/MS method used the same instrument configuration and connected a capillary column (Ultra Alloy5 ; 0.25 um film thickness, 30.0 m length, 0.25 mm ID). Peat samples were weighed as in EGA-MS. The multi-step temperature increase was set to five steps (1) 150-225° C, (2) 225-300° C, (3) 300-375° C, (4) 375-450° C, and (5) 450-600° C for the heart-cut analysis method. The gases generated in each temperature fraction were collected cold by liquid nitrogen in a Cryo-trap and introduced into the GC/MS. Compounds were identified using the NIST20 library database.

RESULTS & DISCUSSIONS

EGA analysis of the vertical sample in SCL19-15 showed that m/z 60, 84, and 114 decreased toward the lower layers in the peak around 300° C, and m/z 83, 85, and 91 increased toward the lower layers in the peak around 430° C. The decrease in the readily degradable component and increase in the persistent component indicated the progress of humification. The decrease in readily degradable components and increase in persistent components indicate the progression of humification. The evaluation method of the four derived components (A, B, C, and D) facilitated the understanding of humification.

Evaluating the EGA method with 5 fractions of temperature range and comparing the loss fractions (2) and (4) by ms-Py-GC/MS method, it was found that the results of component evaluation obtained by EGA method and by ms-Py-GC/MS method are almost the same results.

Peat samples collected from a wide area of Scotland could be shown to have specific chemical characteristics for each region of origin. The western, northern, and southern regions each exhibited similar characteristics.



Characterization of peat samples by collection area using the EGA method