Geographical origin of Thai Hom Mali rice based on elemental and stable isotopic compositions

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Abstract. Thai Hom Mali rice is an economic crop of Thailand and well-known as a premium rice in the world trade. In this research, Thai Hom Mali rice samples were collected from the paddy fields in the northern and northeastern regions of Thailand for verifying the authenticity of rice origin. Multi-element and stable isotopic compositions in rice samples were determined by inductively coupled plasma mass spectrometry (ICP-MS) and isotope ratio mass spectrometry (IRMS), respectively. A radar plot was applied to discriminate the origin of rice. Thai Hom Mali Rice from both regions had similar pattern of isotopic composition. The two regions clearly displayed different distribution of elemental patterns. Therefore, Thai Hom Mali rice can be differentiated from each region using multi-element combined with a radar plot.

1. Introduction
Rice (Oryza sativa) is a staple food for Thai and a large part of the world’s population. Rice is the most important economic crop of Thailand. There are many varieties of rice cultivated throughout the country of Thailand. Thai Hom Mali rice or Thai jasmine rice is well known for Thai rice and worldwide. The best tasting and best quality of Thai Hom Mali rice production has been cultivated in the northeastern region and some area of the northern region of Thailand. It is premium rice in the world trade therefore it is vulnerable to adulteration or mislabeling. Unscrupulous producers attempt to increase profits by mislabeling inferior rice cultivated outside these regions. The rice variety and cultivation area are important factors in market price of rice [1]. The determination of rice authenticity is essential to prevent the mislabeling of rice origin. The elemental and isotopic compositions have been used to investigate the authenticity of rice origin. Yasui and Shindoh [2] reported the distinction of the geographical origin of Japanese brown rice samples by their trace element concentrations analyzed by ICP-AES and ICP-MS. Cluster analysis (Ward method) and PCA using nine elements (Mn, Zn, Fe, Cu, Rb, Mo, Ba, Sr, Ni) could distinguish rice samples cultivated in the Tohoku/Kanto area from those in Hokuriku, and those in Tohoku from those in Kanto. Determination of the geographic origin of brown rice with isotope ratios of $^{11}\text{B}/^{10}\text{B}$ and $^{87}\text{Sr}/^{86}\text{Sr}$ was carried out by Oda, Kawasaki and Hirata [3]. Rice samples collected from 44 sites in Japan, 4 in China, 3 in Australia, 1 in Vietnam and 1 in California were studied for isotope ratios of $^{87}\text{Sr}/^{86}\text{Sr}$ determined by MC-ICP-MS and $^{11}\text{B}/^{10}\text{B}$ determined by ICP-QMS. They found that $^{11}\text{B}/^{10}\text{B}$ and $^{87}\text{Sr}/^{86}\text{Sr}$ ratios could distinguish
rice samples in Japan (\(^{11}\)B/\(^{10}\)B : 3.97-4.13 and \(^{87}\)Sr/\(^{86}\)Sr: 0.706-0.709) from samples in Australia (\(^{11}\)B/\(^{10}\)B : >4.10 and \(^{87}\)Sr/\(^{86}\)Sr: >0.714), samples in China and Vietnam (\(^{11}\)B/\(^{10}\)B : 4.02-4.05 and \(^{87}\)Sr/\(^{86}\)Sr: 0.710-0.711) and sample in California (\(^{11}\)B/\(^{10}\)B : 4.09 and \(^{87}\)Sr/\(^{86}\)Sr: 0.706). Kelly et al [4] studied the geographical origin of premium long grain rice cultivated in USA, Europe, India, and Pakistan using IRMS and ICP-MS. Nine key parameters (C-13, O-18, B, Ho, Gd, Mg, Rb, Se and W) were identified by canonical discriminant analysis and provided 100% discrimination between rice samples from these regions. Kawasaki, Oda and Hirata [5] investigated the strontium isotope ratio (\(^{87}\)Sr/\(^{86}\)Sr) of brown rice cultivated in Australia, California, China and Vietnam by multiple collector inductively coupled plasma mass spectrometry (MC-ICP-MS). The Sr isotope ratios of the Chinese and Vietnamese rice samples (0.710 to 0.711) were slightly higher than those of almost all the Japanese samples (0.706 to 0.709). Australian rice showed the highest Sr isotope ratio (0.715 to 0.717) among all the rice samples. The ratio of Californian rice (0.706) appeared to be lower than that of Japanese rice. This study clearly demonstrated that the Sr isotope ratios could provide the information for the estimation of rice provenance. Suzuki et al [1] determined C and N contents and isotopic compositions (\(\delta^{13}\)C, \(\delta^{15}\)N, and \(\delta^{18}\)O) of polished Koshihikari rice cultivated in Australia, Japan and USA in order to discriminate the geographical origin. The pentagonal radar plots based on the elemental and isotopic compositions are clearly distinguished these cultivation areas. Trace elements in rice from different geographical origins were determined by ICP-OES investigated by Gonzálvez, Armenta and Guardia [6]. “Arròs de València” PDO rice samples have been differentiated from Spanish rice like “Arroz del Delta del Ebro”, “Arroz de Calasparra”, rice from Extremadura and Indian, Japanese and Brazilian ones by using the trace elemental profile and LDA, enabling a 91.30% correct classification for the validation set. Cheajesadagul et al [7] studied the discrimination of geographical origin of rice based on multi-element fingerprinting by high resolution inductively coupled plasma mass spectrometry. A total of 31 Thai jasmine rice and 5 foreign (France, India, Italy, Japan and Pakistan) rice samples were analyzed for multi-elements. Thai jasmine rice can be differentiated from foreign rice samples by radar plots based on six elements (B, Co, Sr, Mo, Rb and Se) and multivariate data analysis (PCA and DA). Furthermore, the DA can differentiate Thai jasmine rice samples according to each region of origin (northern, northeastern or central regions of Thailand). Stable isotopic compositions including C, N, H, O and S, and heavy isotopes of Sr and B have been investigated for tracing the agro-product by Zhao et al [8]. Wu et al [9] studied the geographical origin of cereal grains using element analyser-stable isotope ratio mass spectrometry (EA-SIRMS). Different varieties of rice grown in various countries including Australia, China P.R., France, India, Italy, Japan, Korea, Malaysia, Myanmar, Pakistan, Spain, Taiwan, Thailand, USA and Vietnam analyzed by isotope ratio mass spectrometry and inductively coupled plasma mass spectrometry have been investigate by Li et al [10]. Chung et al [11] investigated the geographic authentication of Asian rice using multi-elemental and stable isotopic data combined with multivariate analysis. Stable isotopic composition and elemental concentration have been applied for tracing the origin of rice in China and Southeast Asia studied by Liu et al [12].

In this work, Thai Hom Mali rice samples were collected from the paddy fields in the northern and northeastern regions of Thailand. The elemental concentration (As, Cd, Cu, Mo and Sr) in the polished rice (or white rice) samples was analyzed by inductively coupled plasma mass spectrometry (ICP-MS). The isotopic composition (\(\delta^{13}\)C, \(\delta^{15}\)N and \(\delta^{18}\)O) was measured by isotope ratio mass spectrometry (IRMS). The discrimination of rice origin was investigated using a radar plot based on elemental and isotopic components.

2. Materials and Methods

2.1. Reagents and standards

Nitric acid 65% (supra-pure analytical grade), hydrofluoric acid 48% (analytical grade) and hydrogen peroxide 30% (analytical grade) were purchased from Merck (Darmstadt, Germany). All solutions were prepared using ultrapure water (>18 MΩ cm) obtained with a Milli-Q water purification system.
The standard solution was purchased from Agilent Technologies (New Haven, CT, USA). The standard reference material used to check the ICP-MS method was rice flour (NIST 1568a) from National Institute of Standards and Technology (NIST; MD, USA). Standard reference materials of IAEA 601 (Benzoic Acid), IAEA 600 (Caffeine), USGS-35 (Sodium Nitrate) and USGS-43 (Indian human hair) were supplied by the International Atomic Energy Agency (IAEA; Vienna, Austria). Laboratory reference materials of Alfalfa Flour, Nylon 6, Bovine Liver, Enriched Alanine and Cellulose were used for quality control.

2.2. Sample collection and preparation
A total of 50 Thai Hom Mali rice samples were obtained from the paddy field in the northern part (Chiang Mai and Chiang Rai) and northeastern region (Roi Et, Si Sa Ket and Yasothon). Ten samples were collected from each cultivation area. The rice samples were air-dried at room temperature. All samples were dehulled to be polished rice or white rice and were ground by a mortar to obtain fine powder. The rice powders were dried again in an oven at 60 ± 2 °C until constant weight, kept in polyethylene containers and stored in a desiccator until analysis.

2.3. Elemental analysis by ICP-MS
All powdered rice samples (0.5 g) were digested in PTFE vessels with 10 mL of 65% w/w HNO₃ and 4 mL of 48% w/w HF on a hot plate at 120 °C for 5 h. After digestion, the samples were evaporated to dryness and the residues were dissolved in 65% w/w HNO₃ and 30% w/w H₂O₂. After drying, the residues were dissolved with 2% v/v HNO₃. Finally, the dissolution samples were diluted to an appropriate concentration for ICP-MS analysis. All sample digestions were completed in triplicate. The standard reference material of rice flour (NIST 1568a) was used to check the procedure accuracy.

The elemental concentrations (As, Cd, Cu, Mo and Sr) in rice samples were determined by ICP-MS (Agilent 7900, Agilent Technologies, USA). The ICP-MS operating conditions for the determination of elements were as follows: RF power, 1550 W; carrier gas flow rate, 0.8 L min⁻¹; makeup gas flow rate, 0.2 L min⁻¹; nebulizer pump, 0.1 rpm; nebulizer, Mira Mist; spray chamber, Scott type; Sampling cone and skimmer cone, Nickle. Rhodium (Rh) was used as an internal standard to compensate for any changes in analytical signals during the operation of ICP-MS. Standard solutions prepared from Agilent multi-element solution were used to create the standard calibration with minimum 5 points.

2.4. Stable isotope measurement by IRMS
Dried rice (20 g) was ground to a fine powder before analysis. For carbon and nitrogen, the powdered rice was weighed (3-4 mg) into a tin capsule. Then, each sample was analyzed by elemental analyzer/isotope ratio mass spectrometry (EA/IRMS) using an Elementar Vario EL Cube or Micro Cube elemental analyzer (Elementar Analysensysteme GmbH, Hanau, Germany) interfaced to either an Isoprime Vision IRMS (Isoprime Ltd., Stockport, UK, a unit of Elementar Analysensysteme GmbH, Hanau, Germany). The final delta values were expressed relative to international standards Vienna Pee Dee Belemnite (VPDB) and Air for carbon and nitrogen, respectively.

For oxygen, the powdered rice was weighed (0.3-0.4 mg) into a silver capsule. Then, oxygen isotope analysis was carried out by thermochemical conversion EA/IRMS (TCEA/IRMS) using an elementar PyroCube (Elementar Analysensysteme GmbH, Hanau, Germany) interfaced to an Isoprime Vision (Isoprime Ltd., Stockport, UK, a unit of Elementar Analysensysteme GmbH, Hanau, Germany). The final ¹⁸O delta values are expressed relative to international standard Vienna Standard Mean Ocean Water (VSMOW).

The isotopic composition is reported as δ, expressed in conventional δ-notation with the unit of parts per thousand or per mil (‰), shown in equation (1) [13].

\[
\delta (\text{‰}) = \left( \frac{R_{\text{sample}}}{R_{\text{standard}}} - 1 \right) \times 1000
\]  

(1)
where \( R_{\text{sample}} \) is the isotope ratio (i.e., \(^{13}\text{C}/^{12}\text{C}, {^{15}\text{N}}/{^{14}\text{N}} \) and \(^{18}\text{O}/^{16}\text{O}\) of the sample, and \( R_{\text{standard}} \) is the isotope ratio of the international standards: for carbon: VPDB; for nitrogen: Air; and for oxygen: VSMOW.

### 2.5 Statistical analysis

The statistical analysis of Microsoft Office Excel 2016 was used to calculate the mean, standard deviation (SD), minimum and maximum. The difference between the means of two data sets was evaluated using the Student’s \( t \)-test at the 95% confidence interval (\( t \)-test; \( p < 0.05 \)).

### 3. Results and Discussion

#### 3.1. Elemental concentration in rice samples by ICP-MS

According to the IUPAC recommendation [14], the limit of detection (LOD) for all elements determined by ICP-MS was estimated from three times the standard deviation of the blank measurements. The limit of detection (in mg kg\(^{-1}\)) obtained from LOD (in \( \mu \)g L\(^{-1}\)) multiplied by the overall dilution factor were as follows: As (0.003), Cd (0.001), Cu (0.23), Mo (0.01) and Sr (0.02), which is adequate for the determination of these elements in rice sample.

The standard reference material of rice flour (NIST 1568a) was used to validate the analytical procedure. The result of analyzed value showed good agreement with the certified value. The recoveries ranged from 94% to 101%.

Fifty Thai Hom Mali rice samples were digested by acid and determined for As, Cd, Cu, Mo and Sr concentrations by ICP-MS. The summary statistics of elemental concentrations in rice samples according to their cultivation provinces were appeared in Table 1. The mean concentration of As was highest in rice sample from Si Sa Ket and lowest in rice sample from Chiang Rai. Rice samples from the northern area (Chiang Mai and Chiang Rai) have rather high concentration of Cd. However, the concentration of Sr in rice samples from the northern part is lower than the northeastern region (Roi Et, Si Sa Ket and Yasothon). All rice samples have similar level of Cu and Mo (\( t \)-test; \( p < 0.05 \)).

The data of As, Cd, Cu, Mo and Sr in rice samples from this work were compared with the previous data reported by Cheajesadagule et al [7] and Srinuttrakul et al [15] shown in Table 2. The concentrations of Cd, Cu, Mo and Sr from this work were higher than those of the previous data. No significant difference was observed for As content between this study and the previous reports (\( t \)-test; \( p < 0.05 \)).

#### 3.2. Stable isotopic composition in rice samples by IRMS

The accuracy of the method was validated by the analysis of standard reference materials. The result obtained from the experiment agreed well with the certified values. The recoveries ranged from 99% to 100%. The precision (± standard deviation) of below ± 0.1 - 0.3‰ was determined for \( \delta^{13}\text{C}, \delta^{15}\text{N} \) and \( \delta^{18}\text{O} \) measurements.

Isotopic compositions of Thai Hom Mali rice according to cultivation area analyzed by IRMS were presented in Table 3. Chiang Mai and Chiang Rai have the same range of isotopic compositions (\( t \)-test; \( p < 0.05 \)). \( \delta^{13}\text{C}, \delta^{15}\text{N} \) and \( \delta^{18}\text{O} \) of rice samples from the northern region were -28.52 to -27.18‰, +3.77 to +6.26‰ and +21.98 to +23.71‰, respectively. Rice samples from the northeastern area showed \( \delta^{13}\text{C}, \delta^{15}\text{N} \) and \( \delta^{18}\text{O} \) values varying from -27.72 to -26.31‰, +1.87 to +9.10‰ and +23.44 to +26.67‰, respectively.

All rice samples have \( \delta^{13}\text{C} \) in the range of C-3 plants (-22‰ to -33‰) [9]. Rice samples from the northeastern region have \( \delta^{18}\text{O} \) value higher than those from the northern area. In general, oxygen isotopic composition of plant materials mainly reflects that of precipitation [1]. The rainfall was used for rice cultivation in both regions, therefore, the precipitation during the rice cultivation period in the northeastern part was greater than that in the northern region. The variation of \( \delta^{15}\text{N} \) of rice sample from the northeastern area (+1.87 to +9.10‰) was broader than those from the northern part (+3.77 to +6.26‰). The isotopic composition of nitrogen in rice depends on soil nutrition especially fertilizer
used for rice cultivation. Generally, organic fertilizers increase $^{15}$N content in soil and plants, whereas the utilization of artificial fertilizers decreases it [16-18]. In comparison with $\delta^{15}$N value of rice from Australia (+9.0‰) using the organic fertilizer reported by Suzuki et al [1], it indicated that one sample from Si Sa Ket (Northeastern region) with $\delta^{15}$N of +9.10‰ was cultivated as organic rice. The chemical fertilizer was used for other rice samples.

**Table 1.** Summary statistics of elemental concentrations in rice samples determined by ICP-MS.

| Region/Province         | As (mg kg$^{-1}$) | Cd (mg kg$^{-1}$) | Cu (mg kg$^{-1}$) | Mo (mg kg$^{-1}$) | Sr (mg kg$^{-1}$) |
|-------------------------|------------------|------------------|------------------|------------------|------------------|
| **North/Chiang Mai**    |                  |                  |                  |                  |                  |
| Mean                    | 0.077            | 0.112            | 1.376            | 0.469            | 0.074            |
| SD                      | 0.025            | 0.065            | 0.146            | 0.145            | 0.014            |
| Minimum                 | 0.050            | 0.032            | 1.225            | 0.325            | 0.057            |
| Maximum                 | 0.144            | 0.182            | 1.533            | 0.703            | 0.091            |
| Median                  | 0.077            | 0.091            | 1.439            | 0.538            | 0.068            |
| **North/Chiang Rai**    |                  |                  |                  |                  |                  |
| Mean                    | 0.059            | 0.079            | 1.675            | 0.440            | 0.072            |
| SD                      | 0.021            | 0.067            | 0.347            | 0.149            | 0.017            |
| Minimum                 | 0.023            | 0.016            | 1.380            | 0.186            | 0.059            |
| Maximum                 | 0.074            | 0.190            | 2.225            | 0.542            | 0.098            |
| Median                  | 0.072            | 0.017            | 1.417            | 0.452            | 0.072            |
| **Northeast/Roi Et**    |                  |                  |                  |                  |                  |
| Mean                    | 0.136            | 0.011            | 1.277            | 0.504            | 0.319            |
| SD                      | 0.056            | 0.017            | 0.602            | 0.217            | 0.103            |
| Minimum                 | 0.048            | 0.001            | 0.613            | 0.171            | 0.182            |
| Maximum                 | 0.215            | 0.053            | 2.533            | 0.878            | 0.521            |
| Median                  | 0.132            | 0.004            | 1.101            | 0.483            | 0.314            |
| **Northeast/Si Sa Ket** |                  |                  |                  |                  |                  |
| Mean                    | 0.145            | 0.012            | 1.916            | 0.593            | 0.351            |
| SD                      | 0.056            | 0.014            | 0.553            | 0.292            | 0.108            |
| Minimum                 | 0.041            | 0.002            | 1.219            | 0.214            | 0.231            |
| Maximum                 | 0.227            | 0.048            | 2.740            | 1.017            | 0.501            |
| Median                  | 0.133            | 0.007            | 1.836            | 0.623            | 0.312            |
| **Northeast/Yasothon**  |                  |                  |                  |                  |                  |
| Mean                    | 0.094            | 0.028            | 1.966            | 0.575            | 0.364            |
| SD                      | 0.033            | 0.032            | 0.940            | 0.139            | 0.215            |
| Minimum                 | 0.054            | 0.003            | 0.552            | 0.276            | 0.107            |
| Maximum                 | 0.166            | 0.110            | 3.278            | 0.744            | 0.825            |
| Median                  | 0.090            | 0.019            | 2.264            | 0.595            | 0.313            |

**Table 2.** Elemental concentration in rice samples from this study and the previous reports.

| Element | This study, $(n = 50)$ | Cheajesadagul et al [7], $(n = 31)$ | Srinuttrakul et al [14], $(n = 30)$ |
|---------|-----------------------|------------------------------------|------------------------------------|
| As      | $0.106 \pm 0.049$     | $0.101 \pm 0.001$                  | $0.116 \pm 0.053$                  |
| Cd      | $0.049 \pm 0.081$     | $0.012 \pm 0.000$                  | $0.027 \pm 0.031$                  |
| Cu      | $1.625 \pm 0.633$     | $1.24 \pm 0.02$                    | $1.158 \pm 0.507$                  |
| Mo      | $0.526 \pm 0.192$     | $0.48 \pm 0.01$                    | No data                            |
| Sr      | $0.234 \pm 0.178$     | $0.141 \pm 0.002$                  | No data                            |
Table 3. Summary statistics of isotopic composition in rice samples analyzed by IRMS

| Region/Province          | δ\(^{13}\)C (‰) | δ\(^{15}\)N (‰) | δ\(^{18}\)O (‰) |
|--------------------------|------------------|------------------|-----------------|
| **North/Chiang Mai**     |                  |                  |                 |
| Mean                     | -27.51           | 4.25             | 23.79           |
| SD                       | 0.22             | 0.96             | 0.80            |
| Minimum                  | -28.52           | 3.77             | 21.98           |
| Maximum                  | -27.18           | 6.26             | 23.71           |
| Median                   | -27.51           | 4.17             | 23.82           |
| **North/Chiang Rai**     |                  |                  |                 |
| Mean                     | -27.61           | 4.87             | 23.05           |
| SD                       | 0.41             | 0.82             | 0.47            |
| Minimum                  | -28.52           | 3.77             | 21.98           |
| Maximum                  | -27.18           | 6.26             | 23.71           |
| Median                   | -27.48           | 4.61             | 23.08           |
| **Northeast/Roi Et**     |                  |                  |                 |
| Mean                     | -27.23           | 4.12             | 25.67           |
| SD                       | 0.21             | 0.84             | 0.80            |
| Minimum                  | -27.59           | 2.99             | 24.52           |
| Maximum                  | -27.01           | 5.65             | 26.67           |
| Median                   | -27.04           | 4.51             | 25.00           |
| **Northeast/Si Sa Ket**  |                  |                  |                 |
| Mean                     | -27.15           | 4.98             | 24.77           |
| SD                       | 0.43             | 1.95             | 0.70            |
| Minimum                  | -27.70           | 2.55             | 23.44           |
| Maximum                  | -26.31           | 9.10             | 25.51           |
| Median                   | -27.03           | 3.68             | 25.13           |
| **Northeast/Yasothon**   |                  |                  |                 |
| Mean                     | -27.33           | 3.76             | 25.79           |
| SD                       | 0.21             | 1.08             | 0.62            |
| Minimum                  | -27.72           | 1.87             | 24.69           |
| Maximum                  | -27.11           | 5.26             | 26.60           |
| Median                   | -27.19           | 4.44             | 26.45           |

3.3. Discrimination of the geographical origin of rice samples by radar plot

The elemental and isotopic concentrations were used to determine the geographical origin of rice. It is difficult to use only one variable to differentiate the rice origin. Therefore, in this study a radar plot was applied to classify the geographical origins of rice. A radar plot based on the elemental contents of As, Cd, Cu, Mo and Sr was used to discriminate the cultivation origin between the northern and northeastern regions. The distribution of elemental pattern in Figure 1 showed the different characteristics for both regions. Therefore, a radar plot based on the elemental concentration could clearly discriminate the origin of Thai Hom Mali rice from each region.

A radar plot based on the isotopic compositions of δ\(^{13}\)C, δ\(^{15}\)N and δ\(^{18}\)O and the contents of C and N was used to distinguish the geographical origin of rice. Both regions had similar pattern of isotopic composition displayed in Figure 2. Although it is difficult to discriminate the regional origins of rice in Thailand by isotopic composition, these data would be useful for comparison with those of other countries.
Figure 1. Radar plots showing the geographical origin of Thai Hom Mali rice samples based on the concentrations of As, Cd, Cu, Mo and Sr (a) Northern and (b) Northeastern regions.

Figure 2. Radar plots showing the geographical origin of Thai Hom Mali rice samples based on $\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$, C and N (a) Northern and (b) Northeastern regions.

4. Conclusions
The elemental and isotopic compositions in polished Thai Hom Mali rice samples collected from the paddy fields in the northern and northeastern regions of Thailand were carried out by ICP-MS and IRMS, respectively. The developed methods are reliable to determine the elemental and isotopic compositions in rice sample. The elemental data is more useful than isotopic values to characterize the geographical origin of Thai Hom Mali rice. Geographical origin of Thai Hom Mali rice could be discriminated based on elemental concentration combination with a radar plot. The obtained results would be useful for a database of Thai Hom Mali rice using for the determination of its geographical origin.
Acknowledgments
We would like thank the staff of Agricultural Extension Office for their assistance in cooperation with the farmers. We wish to express our thanks to the farmers for providing their rice samples. This work has been financially supported by the International Atomic Energy Agency (Research Contract No. 16523).

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