

# APPLICATION OF STABLE ISOTOPE SIGNATURES IN FOOD TRACEABILITY

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Stable isotope analysis has widely been used to trace the origin of organic materials in various fields, such as geochemistry, biochemistry, archeology and petroleum. In past a decade, it has also become an important tool for food traceability study. The globalisation of food markets and the relative ease with which food commodities are transported through and between countries and continents, means that consumers are increasingly concerned about the origin of the foods they eat. The natural abundance isotope variation such as carbon, nitrogen, hydrogen and oxygen are use as geographic tracers or marker to determine the geographic origin of fruits, crop, vegetables and food products from animal. The isotopic compositions of plant materials reflect various factors such as isotopic compositions of source materials and their assimilation processes as well as growth environments. This paper will discuss on stable carbon and nitrogen isotopic compositions in rice, advantages, limitations and potential of other analysis applications that can be incorporated in food traceability system.

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## INTRODUCTION

Food has generated multi-million profit industries. The globalisation of food markets and the relative ease with which food commodities are transported through

and between countries and continents, means that consumers are increasingly concerned about the origin of the foods they eat whether it is natural or processed food. The safety, quality and authenticity of food are becoming focal issue for all faction along the supply chain including farmers, traders and consumers. Traceability systems play a key role in assuring food safety, help to prove authenticity, to combat fraudulent practices, and to control adulteration, which are important issues for economic, religious or cultural reasons. Research to date covers a wide range of food commodity and premium products including to wine, cheese, meat, honey, coffee and olive oil [1].

Stable isotope analysis has widely been used to trace the origin of organic materials in various fields, such as geochemistry, biochemistry, archeology and petroleum [2,3]. In recent years, application of stable isotopic measurement technique had been integrate in food traceability study along with other analytical approaches including quantification of elemental compositions, concentrations of fatty acids and quantification of rare earth elements [4]. The natural abundance isotope variation such as carbon, nitrogen, hydrogen and oxygen are use as geographic tracers or marker to determine the geographic origin of fruits, crop, vegetables and food products from animal. The isotopic compositions of plant materials reflect various factors such as isotopic compositions of source materials (water, fertilizer and gas) and their assimilation processes as well as growth environments particularly the climate and altitude [5].

In this work, we determined stable carbon and nitrogen isotopic compositions of rice from 3 different regions by using isotope ration mass spectrometry. The study will highlight the potential of stable isotopic composition in discriminate cultivation areas of rice and discuss of any limitation as well as possible integration of other analytical approaches.

## METHODOLOGY

### Samples collection and preparation

Samples of rice were collected from MADA area including Kedah and Perlis ( $n = 50$ ) and Selangor ( $n = 20$ ). All samples were dried and ground to fine powder prior analysis. 27 samples of rice from China were obtained through exchange program under project IAEA RAS5062 *Building Technological Capability for Food Traceability and Food Safety Control System Through The Use of Nuclear Analytical Techniques*.

### Isotope ratio determination

Samples for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  analysis were prepared by weighing triplicate 1mg into small tin capsules (3mm x 2 mm x 5mm). Then the capsules were folded and compressed to contain the sample and minimize any air present. Nitrogen and carbon isotopes were introduced through the autosampler into SerCon ANCA-GSL Elemental Analyzer preparation line via combustion process before being analysed using SerCon GEO 2020 Continuous Flow Isotope Ratio Mass Spectrometer (IRMS). The stable isotopic compositions were recorded in the delta ( $\delta$ ) notation relative to a standard: Vienna Pee Dee Belemnite (VPDB) for carbon and atmospheric nitrogen for nitrogen. Analytical performance was checked by placing laboratory standard between samples to check for stability and to allow drift correction when necessary. The IA-R001 Wheat Flour (Iso-Analytical, UK) was applied as laboratory standard with certified  $\delta^{13}\text{C} = -26.43\text{‰}$  and  $\delta^{15}\text{N} = 2.55\text{‰}$ . The typical precision for analysis of control material is  $\pm 0.1\text{‰}$  for  $\delta^{13}\text{C}$  and  $\pm 0.3\text{‰}$  for  $\delta^{15}\text{N}$ .

## RESULTS AND DISCUSSION

The  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  of rice from 3 locations were shown in Fig.1. The  $\delta^{13}\text{C}$  values of rice from Kedah, Selangor and China ranged from  $-28.21\text{‰}$  to  $-26.02\text{‰}$ ,  $-28.99\text{‰}$  to  $-27.38\text{‰}$  and  $-31.49\text{‰}$  to  $-23.78\text{‰}$ , respectively. Rice and some other plants such as wheat, rye, cotton and flowering plants have  $\delta^{13}\text{C}$  values varying from  $-22$  to  $-35\text{‰}$  [6]. China rice had shown wide range of  $\delta^{13}\text{C}$  values compared to Kedah and Selangor. Previous study on rice had been conducted by several researches elsewhere. The  $\delta^{13}\text{C}$  values of 160 rice samples from different counties in Guangdong province of China ranged from  $-29.086\text{‰}$  to  $-27.787\text{‰}$  and the average of those was  $-28.377\text{‰}$ . Past study had shown that the mean  $\delta^{13}\text{C}$  value of rice from India and Pakistan was  $-27.4\text{‰}$ . Meanwhile, the American and European rice samples were  $-26.2\text{‰}$  and  $-25.5\text{‰}$  [7].

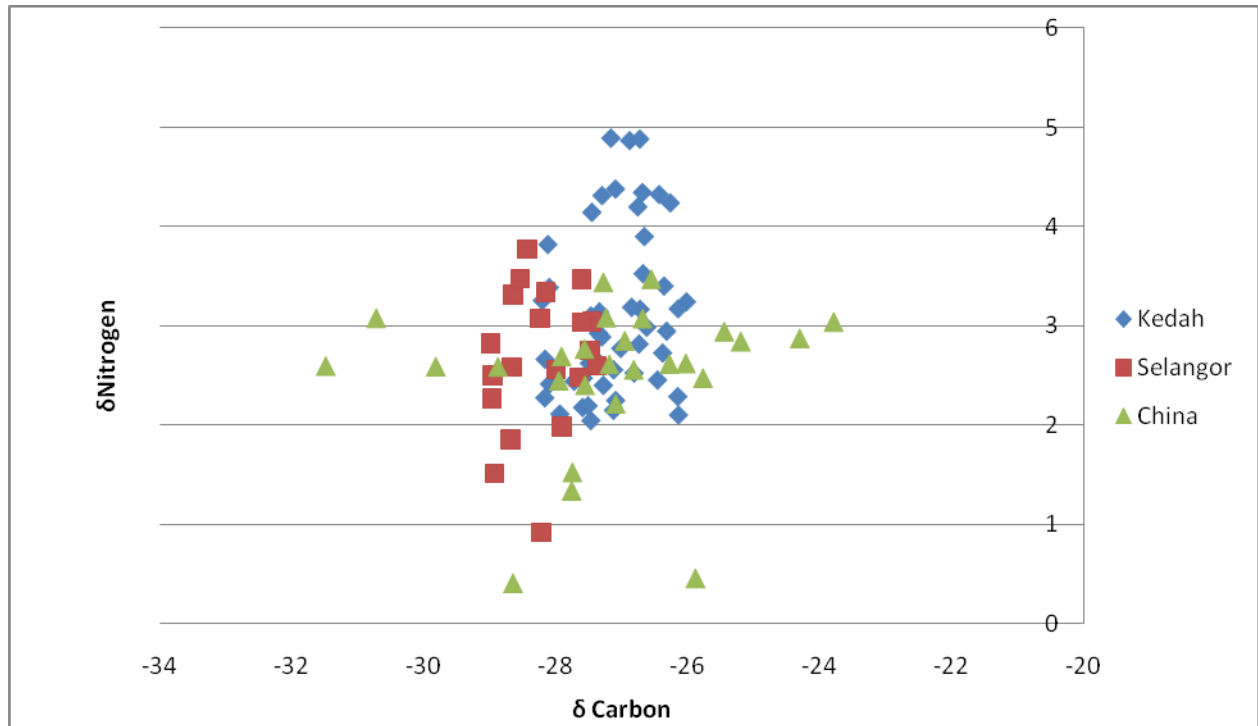


Fig. 1. Two-dimensional distribution of  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values in rice from Kedah, Selangor and China.

For all rice samples, the carbon isotopic compositions were consistent with those of general plant materials however the differ of value may not only due in term of rice variety but a small difference in growth condition such as temperature, rainfall and hours of sunlight probably reflected in the factors on the  $\delta^{13}\text{C}$  values [8]. Previously, an investigation had proved that the  $^{13}\text{C}$  enrichment in rice is generally attributed to a degree of dryness at the cultivating environment: when a plant grows in the more arid condition, the  $\delta^{13}\text{C}$  of the plant becomes heavier [9]. Annually dry season is encountered by the northern part of Peninsular Malaysia that includes the cultivation area of rice in Perlis. Therefore, some of the rice samples in Kedah were apparently heavier in  $\delta^{13}\text{C}$  value than Selangor sample. Meanwhile, China is a large country with enormously varies the climate conditions and it demonstrate over the wide range of  $\delta^{13}\text{C}$  value of those China rice samples.

The  $\delta^{15}\text{N}$  value of rice Kedah, Selangor and China ranged from 1.27‰ to 4.90‰, 0.92‰ to 3.77‰ and 0.41‰ to 3.47‰, respectively. Previous study on Japanese rice indicated the  $\delta^{15}\text{N}$  value ranged from 0.4‰ to 6.5‰ [10]. Nitrogen isotopic composition of rice is thought to depend mainly on soil nutrition, where the rice is cultivated. Organic fertilizers increase  $^{15}\text{N}$  content in soil and plants whereas the utilization of artificial fertilizers decreases it [11]. Most of the rice from China,

Kedah and Selangor grown using artificial fertilizer that indicated by lower  $\delta^{15}\text{N}$  values ( $\delta^{15}\text{N} < 4\text{‰}$ ). Generally, nitrogen fraction in nature are due to kinetic effects of non-biological and biological effect. There are two non-biological fraction effects, dissolution in water and diffusion in water. Biological effect by bacteria display several fractions process: nitrification, denitrification and nitrogen fixation [6]. Therefore, agricultural practice somehow contribute to isotope nitrogen compositions in soil environment.

## CONCLUSION

As a summary, the  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  values could explain a difference in climate and source of fertilizer. The observed difference on stable isotopic composition in those 3 regions were slightly significant with a degree of discrimination on the geographic origin. However, further investigation is needed by incorporating the elemental analysis and additional of oxygen, strontium and boron isotopic compositions, to enhance the prediction capabilities in order to discriminate geographical origin of rice. Information from both analytical approaches have to develop in parallel with chemometrics (multivariate statistic) that provide a powerful tool for food traceability study particularly of high demand and premium food.

## ACKNOWLEDGEMENTS

This work was supported by the Scienfund Grant under MOSTI. Author wish to thank all the technical personal who involved in the sampling session and analysis.

## REFERENCES

- [1] A. Gonzalves, S. Armenta, M. de la Guardia. Trace-element composition and stable-isotope ratio for discrimination of foods with Protected Designation of Origin, Trends In Analytical Chemistry, 28:11(2009) 1295-1311.
- [2] Rozanski, K., Araguás-Araguás, L., & Gonfiantini, R. Relation between long term trends of oxygen-18 isotope composition of precipitation and climate. Science, 258 (1992) 981–985.
- [3] IAEA & UNESCO. Environmental isotopes in the hydrological cycle: Principles and applications. Vol1. International Atomic Energy Agency and

United Nations Educational, Scientific and Cultural Organization (2000) Paris/Vienna.

- [4] R. J. McLeod, M. Garland, R. V. Hale, S. Steiman, R. D. Frew. Determining the most effective combination of chemical parameters for differentiating the geographic origin of food products: an example using coffee beans, *J. Food Chem. Nutr.*, 01:02 (2013) 49-61.
- [5] Y. Zhao, B. Zhang, G. Chen, A. Chen, S. Yang, Z. Ye. Recent developments in application of stable isotope analysis on agro-product authenticity and traceability, *Food Chem.*, 145 (2014) 300-305.
- [6] Z. Muccio, G. P. Jackson. Isotope ratio mass spectrometry, *Analyst*, 134 (2009) 213-222.
- [7] S. Kelly, M. Baxter, S. Chapman, C. Rhodes, J. Dennis, P. Brereton. The application of isotopic and elemental analysis to determine the geographical origin of premium long grain rice. *Eur. Food Res. Technol.*, 214 (2002) 72-78.
- [8] Y. Wu, D. Luo, H. Dong, J. Wan, H. Luo, Y. Xian, X. Guo, F. Qin, W. Han, L. Wang, B. Wang, Geographical origin of cereal grains based on element analyser-stable isotope ratio mass spectrometry (EA-SIRMS), *Food Chem.*, 174 (2015) 553–557.
- [9] T. Korenaga, M. Musashi, R. Nakashita, Y. Suzuki. Statistical analysis of rice samples for compositions of multiple light elements (H, C, N and O) and their stable isotopes, *Analytical Science*, 26 (2010) 873-878.
- [10] Y. Suzuki, Y. Chikaraishi, N. O. Ogawa, N. Ohkouchi, T. Korenaga, Geographical origin of polished rice based on multiple element and stable isotope analyses, *Food Chem.*, 109 (2008) 470-475.
- [11] Shearer, G., & Legg, J. O.. Variation in the natural abundance of  $^{15}\text{N}$  of wheat plants in relation to fertilizer nitrogen applications. *Soil Science Society of America Journal*, 39 (1975) 896–901.