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# APPLICATION OF STABLE ISOTOPE SIGNATURES IN FOOD TRACEABILITY

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## *ABSTRACT*

*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 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 of stable isotope variation such as carbon, nitrogen, hydrogen and oxygen are used 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 that been determined by Isotope Ratio Mass Spectrometry, advantages, limitations and potential of other analysis applications that can be incorporated in food traceability system.*

**Keywords:** Food traceability, Isotope Ratio Mass Spectrometry, rice, stable isotope, $\delta^{13}C$, $\delta^{15}N$,

## INTRODUCTION

Food has generated multi-million profit industries. Food commodity supplies that being transported across country and continents arise significant concern among consumers regarding the origin of foods they consume 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 wine, cheese, meat, honey, coffee and olive oil (Gonzalves et al., 2009).

Stable isotope analysis has widely been used to trace the origin of organic materials in various fields, such as geochemistry, biochemistry, archeology and petroleum (IAEA & UNESCO, 2000; Rozanski et al.,1992). 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 (McLeod et al., 2013). The geographic origin of fruits, crop, vegetables and food products from animal can be determined by natural isotope variation such as carbon, nitrogen, hydrogen and oxygen that holding unique signature that eventually use as geographic tracers or maker. The natural characterization of stable isotope in food reflect several factors such as its composition of source materials (water, fertilizer and gas) and their assimilation processes as well as growth environments particularly the climate and altitude (Zhao et al., 2014).

In this work, we determined stable carbon and nitrogen isotopic compositions of rice from 3 different regions by using isotope ratio 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.

## MATERIALS AND METHODS

### 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*. Samples for $\delta^{13}C$ and $\delta^{15}N$ analysis were prepared by weighing triplicate 1mg into small tin capsules (3 mm x 2 mm x 5 mm). Then the capsules were folded and compressed to contain the sample and minimize any air present.

### Isotope Ratio Determination

Sample materials containing 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 and expressed as a per mil (‰) relative to a standard: Vienna Pee Dee Beleminite (VPDB) for carbon and atmospheric nitrogen for nitrogen. Analytical performance was checked by placing laboratory standard between the 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 $\delta^{13}C$ = -26.43‰ and $\delta^{15}N$ = 2.55‰. The typical precision for analysis of control material is ±0.1‰ for $\delta^{13}C$ and ±0.3‰ for $\delta^{15}N$.

## RESULTS AND DISSCUSSION

The $\delta^{13}C$ and $\delta^{15}N$ of rice from 3 locations were shown in Figure1. The $\delta^{13}C$ values of rice from MADA area, Selangor and China ranged from -28.21‰ to -26.02‰, -28.99‰ to -27.38‰ and -31.49‰ to -23.78‰, respectively.. China rice had shown wide range of $\delta^{13}C$ values compared to Kedah and Selangor. Previous study on rice had been conducted by several researches elsewhere. The $\delta^{13}C$ values of 160 rice samples from different counties in

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Guangdong province of China ranged from -29.086‰ to -27.787‰ and the average of those was -28.377‰. Past study had shown that the mean $\delta^{13}C$ value of rice from India and Pakistan was -27.4‰. Meanwhile, the American and European rice samples were -26.2‰ and -25.5‰ respectively (Kelly et al., 2002). Rice and some other plants such as wheat, rye, cotton and flowering plants have $\delta^{13}C$ values varying from -22 to -35‰ (Muccio and Jackson, 2009).

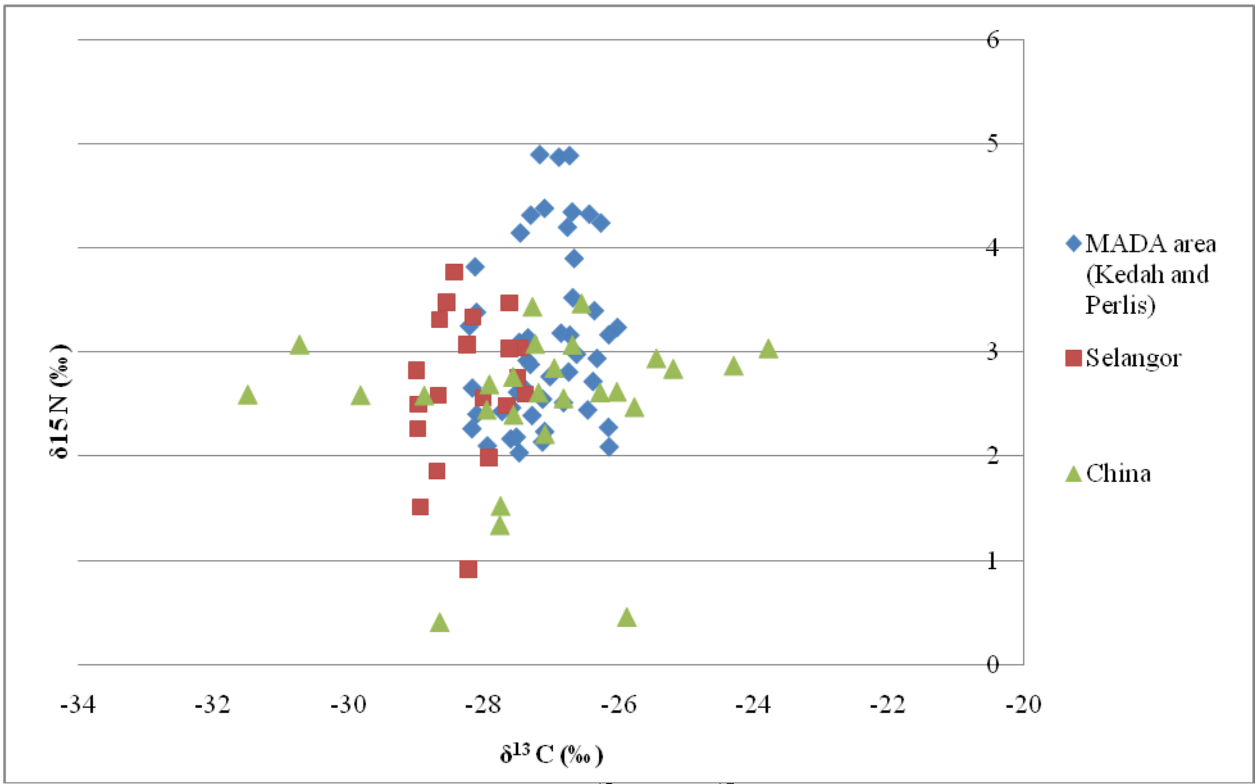


Figure 1: Two-dimentional distribution of $\delta^{13}C$ and $\delta^{15}N$ values in rice from MADA area, Selangor and China

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 $\delta^{13}C$ values (Wu et al., 2015). Previously, an investigation had proved that the $^{13}C$ 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 $\delta^{13}C$ of the plant becomes heavier (Korenaga et al., 2009). Annually dry season is encountered by the northern part of Peninsular Malaysia that includes the cultivation area of rice in Kedah and Perlis. Therefore, some of the rice samples in Kedah and Perlis were apparently heavier in $\delta^{13}C$ value than Selangor sample. Meanwhile, China is a large country with enormously varies the climate conditions and it demonstrate over the wide range of $\delta^{13}C$ value of those China rice samples.

The $\delta^{15}N$ value of rice MADA area, 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 $\delta^{15}N$ value ranged from 0.4‰ to 6.5‰ (Suzuki et al., 2008). Nitrogen isotopic composition of rice is thought to depend mainly on soil nutrition, where the rice is cultivated. Organic fertilizers increase $^{15}N$ content in soil and plants whereas the utilization of artificial fertilizers decreases it (Shearer and Legg, 1975). Most of the rice from China, MADA area and Selangor grown using artificial fertilizer that indicated by lower $\delta^{15}N$ values ($\delta^{15}N$ < 4‰). A few locations in MADA area illustrated in higher $\delta^{15}N$ values ($\delta^{15}N$ > 4‰) that signify the organic fertilizer usage. Generally, nitrogen fraction in nature are due to kinetic effects of non-biological and biological effect. There are two non-biological fration effects, dissolution in water and diffusion in water. Biological effect by bacteria display seversal fractions process: nitrification, denitrification and nitrogen fixiation (Kelly et al., 2002). Therefore, agricaltural pratice somehow contribute to isotope nitrogen compositions in soil environment.

## CONCLUSIONS

As a summary, the $\delta^{13}C$ and $\delta^{15}N$ 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 incorperating 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 taceability study particularly of high demand and premium food.

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