Analysis of Food Dyes in Food – Confirmation of the Presence of Non-Permitted Dyes by LC/MS and Structural Analysis of Unknown Dyes by NMR –

Fusako Ishikawa
Department of Food Safety, Division of Food Additives, Tokyo Metropolitan Institute of Public Health
3-24-1, Hyakunincho, Shinjuku-ku, Tokyo 169-0073, Japan

Summary
For general analysis of food dyes in foods, they are first extracted and cleaned by wool or polyamide dyeing methods, and then determined by thin layer chromatography (TLC) and/or high performance liquid chromatography with a photodiode array detector (PDA-HPLC).
In recent years, liquid chromatography/mass spectrometry (LC/MS) has become increasingly employed for determination of trace levels of pesticides and antibiotics in foods.
In this article, we focus on two areas, determination of non-permitted dyes and structural analysis of unknown dyes in foods, using LC/MS and nuclear magnetic resonance (NMR).

1) In Akasu red vinegar made in Hong Kong, large amounts of Amaranth (R2) and trace levels of Red 2G (R2G), Azo Rubine (Azo), Fast Red E (FRE) and Brilliant Blue FCF (B1) were suggested from the results of TLC and PDA-HPLC. To confirm the results, we optimized LC/MS conditions for determination of these dyes and examined the suspect sample. As a result, the presence of the food dyes at trace levels in Akasu was confirmed, indicating the utility of this approach for detection of dyestuffs in foods.

2) Rose Bengal (R105) and two unknown dyes were furthermore detected in Hajikami, ginger pickles in vinegar, by TLC and PDA-HPLC. We next tried to characterize the unknown dyes by LC/MS and NMR. Judging from results of LC/MS, the dyes were speculated to be R105 with one or two aberrant hydrogens in place of iodine. NMR analysis additionally demonstrated the positions of the substitute hydrogen in these dyes.