

Determination of (D/H)CH₃ of acetic acid molecules by SNIF-NMR method and its application on the adul

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ABSTRACT

Vinegar is widely used as a food condiment in Asian countries. It is common to find fermented vinegar product adulterated with synthetic acetic acid or molasses alcohol vinegar but being alleged pure fermented vinegar in the market. The objective of this study was to evaluate the SNIF-NMR method (site-specific natural isotopic fractionation by nuclear magnetic resonance) for adulteration identification of rice vinegars. Firstly, extractive distillation was used to raise acetic acid concentration. An optimization condition was determined using orthogonal array design, and the effect of extractant and sample concentration on the (D/H)CH₃ value were investigated. Subsequently, detection accuracy for adulterated rice vinegar was evaluated by the SNIF-NMR method. The results obtained were as follows: The optimal condition was to extract acetic acid using a vinegar sample to : extractant ratio at 1 : 1 (v/v) for 15 min and repeated for 7 times. The extraction ratio of acetic acid reached 93.65% for ethyl acetate and 80.75% for ether. It was found that acetic acid concentration could be raised from 5 g/100 mL to 33.84 and 51.65 g/100 mL, respectively. Although the (D/H)CH₃ values in acetic acid solution were not affected by the extractant during the SNIF-NMR determination, apparent differences were observed while acetic acid concentration was below 50 g/100 mL. This indicated that acetic acid concentration was a key factor in the SNIF-NMR method. Almost same (D/H)CH₃ values (98.45-98.62 ppm) for pure rice vinegar (50% in acetic acid concentration) made from three rice varieties Taikeng 9 (TK-9), Taichung sen 10 (TCS-10) and Tainan 11 (TN-11) were obtained. On the other hand, higher values for synthetic acetic acid (131.58 ppm) and molasses alcohol vinegar (108.46 ppm) were confirmed under the same concentration. The (D/H)CH₃ values for rice vinegar were in proportional to the adulteration level of synthetic acetic acid or molasses alcohol vinegar. The linear correlation ($R^2 > 0.97$) indicated that SNIF-NMR method was acceptable for the adulteration identification of pure rice vinegars.

Keywords : extractive distillation、rice vinegar、identification of pure rice vinegar、SNIF-NMR

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