Development of an environmentally more benign reflectometric method for determination of copper(II) ions in sugarcane spirit

João Carlos de Souza[1*](PG), Helena Redigolo Pezza[1] (PQ), Leonardo Pezza[1] (PQ).

**souza_jc33@yahoo.com.br*

[1]Universidade Estadual Paulista "Júlio de Mesquita Filho" – Unesp – Institute of Chemistry Rua Prof. Francisco Degni,55, Jardim Quitandinha, Araraquara-SP, CEP: 14800-060.

Keywords: *spot test, diffuse reflectance spectrometry, copper, sugarcane spirit.*

Introduction

The Brazil increasingly has been highlighted in the production of sugarcane spirit, the second most consumed alcoholic beverage in the country and the third most consumed in the world[1]. The sugarcane spirit is defined as a beverage with an alcohol content 38-54% v/v at 20.0ºC, containing a maximum amount of copper ions allowed from 5.00 mg L^{-1}. [2]

The increased consumption of sugarcane spirit, together with greater exports, requires that the manufacturing process should be based on rigorously controlled practices in order to obtain a standardized product with satisfactory physicochemical and sensory characteristics.

This study describes a simple and more environmentally friendly method by combined spot test-diffuse reflectance spectroscopy for the determination of copper(II) ions in sugarcane spirit.

Results and discussion

The method is based on the reaction between the copper(II) ions with the chromogenic agent 1-(2-pyridylazo)-2-naphthol (PAN) producing a colored compound on the filter paper. With already optimized the experimental conditions, was added 30µL of PAN solution (0.12% m/v in ethanol), after drying, 30µL of the analyte in an alcoholic concentration of 40% v/v in pH = 5.00.

The reflectance measurements were performed at 559 nm in a portable equipment of diffuse reflectance, composed of an integrated sphere with optical fiber and a spectrometer. The ions Cd(II), Co(II), Pb(II), Fe(III), Mn(II), Ni(II) and Zn(II) are interfering in the reaction. This interference is minimized at the level of 2.0% with addition of maskants reagents malonic acid and sodium triphosphate, at the concentration of 0.25% m/v each.

An analytical curve was constructed using the optical density of the reflectance signal (A_R) versus the concentration of copper(II) ions (mg.L^{-1}) and is described by the equation $\Delta A_R = 0.0121\ C_{Cu(II)} + 0.0038$ (r^2= 0.9982). The limits of detection and quantification, calculated relative to the standard deviation of a reagent blank, were 0.12 and 0.47 mg.L^{-1}, respectively. The relative standard deviation (RSD) intraday and interday was 1.82% and 2.64%, respectively, showing good repeatability.

Ten marks of sugarcane spirit were used for the application of the proposed method.

Table 1. Concentrations of copper(II) ions in commercial samples of sugarcane spirit.

Samples	Alcohol content (% v/v)	Region (city/state)	$[Cu^{2+}]$ standard method (mg.L^{-1})	$[Cu^{2+}]$ proposed method (mg.L^{-1})	Test t[a]	Relative Error (%)
1	45	Brotas/SP	2.90 ± 0.04	2.85 ± 0.06	3.0469	-1.72
2	39	Pirassununga/ SP	0.52 ± 0.03	0.54 ± 0.07	0.9818	3.85
3	40	Jaú/SP	4.30 ± 0.06	4.22 ± 0.04	2.3983	-1.86
4	38	Jaú/SP	4.39 ± 0.04	4.33 ± 0.03	2.0591	-1.37
5	39	Santa Rita. P. Quatro/SP	0.99 ± 0.02	0.97 ± 0.02	1.7143	-2.02
6	39	Pirassununga/ SP	0.67 ± 0.04	0.68 ± 0.08	0.3388	1.49
7	39	Rio Claro/SP	1.15 ± 0.02	1.19 ± 0.01	2.5762	3.48
8	38	Pirassununga/ SP	0.46 ± 0.09	0.48 ± 0.09	1.1749	4.35
9	38	Patrocínio Pta./SP	n.d.	n.d.	-	-
10	39	Jundiaí/SP	n.d.	n.d.	-	-

[a]: Test t performed at 95% of confidence, n = 3; $t_{tabulated}$ = 4,303.

The results showed good precision and accuracy compared with the method oficial[4]. None of the analyzed samples showed levels of copper(II) ions above that allowed by brazilian law, showing care and adequate cleaning of the still used in the production of sugarcane spirits.

Conclusions

The proposed method is very attractive for use in routine analysis of copper(II) ions in sugarcane spirit, due to simplicity, economy, speed, sensitivity, using small amounts of reagents and samples, good precision and accuracy, as well as ease of performance and portability (possibility of measures "in situ").

Acknowledgement

Institute of Chemistry of UNESP and CAPES.

[1]SEBRAE-MG; Diagnóstico da cachaça de Minas Gerais. Belo Horizonte, 2011, 259.

[2]MAPA. Ministério da Agricultura, Pecuária e Abastecimento. Instrução normativa n° 13 de 29 de junho de 2005. Diário Oficial da União, Brasília, 30/06/2005.

[3]Lenardão, E. J., Química Nova. 2003, 26(1), 123.

[4]AOAC. Associoation of Official Analytical Chemists Official. Methods of Analysis. 2000, chapter 26, p.7.