

PHYSICAL AND CHEMICAL CHARACTERIZATION OF ETHANOL OBTAINED FROM WASTE COFFEE INDUSTRY

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Keyword: ethanol, biofuel, waste coffee

Introduction

The National Laboratory of Metrology in Costa Rica (LACOMET), along with the National Laboratory of Nanotechnology (LANOTEC) conducted a research on purified and characterized ethanol produced from the waste coming from the coffee industry, in order to establish whether this would comply with chemical and physical parameters to qualify as a biofuel. The results show that the purified ethanol obtained has chemical and physical properties suitable to be classified like a biofuel. These results have national impact in the development of new biofuel sources, because in the past 5 years, Costa Rica has adopted laws and technical regulations for the biofuel production and commercialization.

Methodology

Characterization of raw material. In order to have an initial characterization, the material was measured for the content of Total Solid Dried at 103 °C – 105°C and the Ashes and Volatile Solid Ignited at 550 °C, according with the Standard Methods 2540 B and 2540 E of APHA-AWWA-WEF.



Fig 1. Imagen of the raw material (A), material obtained after a discoloration process (B), material obtained after distillation process (C). The raw material was a crude ethanol obtaining of the fermentation process of the wastewater coffee.

Purification process. The purification was carried out by 3 steps: the first step was the elimination of the suspend solid of the crude ethanol. The filtration process was made with a filter of HVLP 0,22 µm Millex®-GV. The second step is to submit the material to a process of impurities absorption and discoloration. Then, 40 g of activate carbon per 1000 mL of crude ethanol with constant stirring for 24h were added. Finally, the third step is that the ethanol is distilled into a fractional distillation system with a rate flow of 1 mL min⁻¹. The diagram of the purification process can be seen in the Fig 2.

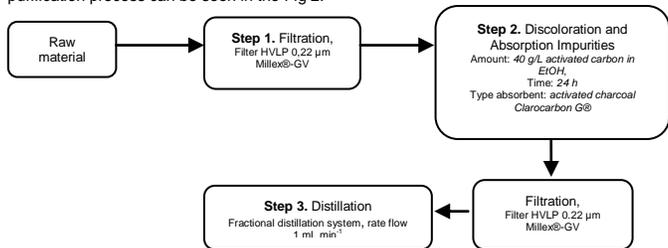


Fig 2. Purification process of crude ethanol obtained from waste coffee industry

Characterization of Ethanol. The characterizations of ethanol consisted in 1 organoleptic, 5 physical and 4 chemical assays: Color (Method 2120 C of the APHA-AWWA-WEF), Density (ASTM D 4052), Refraction Index, Electrolytic conductivity (ASTM D 1125), Surface tension (Du Nouy method), Viscosity, Number acid (ASTM D1613-06), FTIR-ATR and % vol EtOH (OIML, 1975).

Results y discussion

The characterization of raw material (crude ethanol) showed that it has a high level of TSD. This is because the process of fermentation and production of the ethanol from waste coffee is yet not optimized. About 92 % of this TSD was easily volatile material, which ignited at 550°C. This suggests that the majority of the TSD was organic material and only 8 % correspond to ashes, indicated that the raw material have a small portion of inorganic material (see table 1).

Table1. Determination of Total Solid Dried (TSD), Ashes and Volatile Solid (VS) ignited at 550 °C in crude ethanol.

Parameter	Value ^a
TSD (mg L ⁻¹)	498 ± 29
VS (mg L ⁻¹)	456 ± 26
Ashes (mg L ⁻¹)	42 ± 12

^aThe value was reported with the expanded uncertainty, for a coverage factor K=2 for a 95% of confidential interval.

The efficiency of the discoloration process of ethanol was evaluated using a spectrum UV-Vis of the products obtained in every step of purification process.

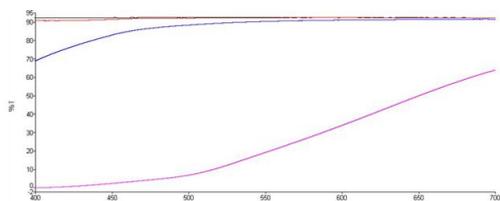


Fig 3. Spectrum UV-Vis of the different products obtained in the purification process. (purple) filtered raw material; (blue) discolored ethanol; (red) Ethanol purified; (black) Commercial Ethanol.

The results in Fig 3, proves that the process of discoloration significantly eliminated the color in the raw material. However, for a total elimination of color is necessary to submit the material through a distilling process.

The effectiveness of the complete purification process can be appreciated in Fig 4. It can be seen that the content of ethanol (%vol.) increased near 23%. Moreover, purified ethanol compared against commercial ethanol have similar concentration.

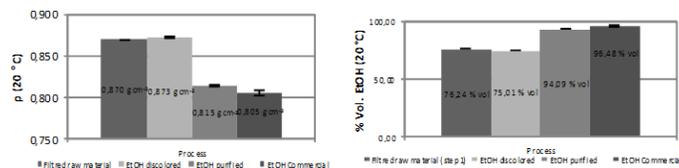


Fig 4. Evaluation of the density and content of EtOH in the purification process. The error bars represent the expanded uncertainty (k=2).

In order to perform the final product identification, a FTIR-ATR of the purified ethanol was performed. The result was compared with the spectrum FTIR-ATR of certified reference material of NIST (Fig. 5).

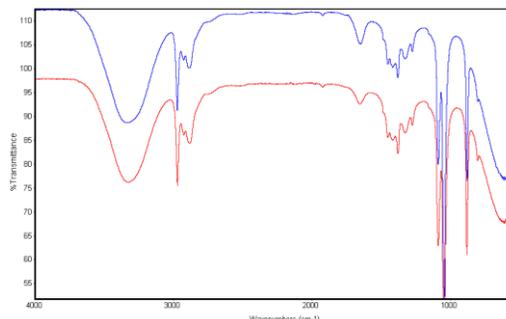


Fig 5. FTIR-ATR of the ethanol obtained from waste coffee (blue) and SRM 2900 Ethanol 95, 6% by mass (red)

The results of the characterization of purified Ethanol are summarized in the table 2. It can be appreciated that the purified Ethanol obtained and from waste coffee has excellent physical and chemical properties to be used as biofuel.

Parameter	Ethanol of Waste Coffee ^a	Biofuel (Hydrated Ethanol) ^b	min/ max
Color	colorless	Colorless or yellow	
Density (kg m ⁻³)	815 ± 2	807,6 - 811	max
Refraction Index (λ = 589 nm)	1,364 ± 0,004	N.E	NE
Electrolytic conductivity (µS m ⁻¹)	3698 ± 10	500	max
Surface tension (mN m ⁻¹)	23,06 ± 0,12	N.E	N.E
Viscosity (cP)	1,61 ± 0,01	N.E	N.E
% vol EtOH	94,09 ± 0,04	92,6 – 93,8	min
Number Acid (as mg L ⁻¹ of CH ₃ COOH)	19,6 ± 0,27	30	max
Involatile material (mg L ⁻¹)	<10	30	max

^aThe value was reported with the expanded uncertainty, for a coverage factor K=2 for a 95% of confidential interval.

^bBrazilian Fuel Ethanol specification of the National Petroleum Agency (Hydrated Ethanol)

^cNE: not specified

Conclusions

We conclude that the purified ethanol obtained of waste coffee industry has chemical and physical properties to be classified like a biofuel. In the next stage of the project will try to do more testing as content of ethanol by GC-FID, purity by DCS, TGA and ionic chromatography, with the objective of better elucidate the properties of this material.

Reference

- APHA-AWWA-WEF. Standard Methods for the Examination of water and Wastewater. 1995, 19th Edition, United State of America.
- ASTM D 4052: "Standard Test Method for Density and Relative Density of Liquids by Digital Density Meter".
- ASTM D-1613: "Standard Test Method for Acidity in Volatile Solvents and Chemicals Intermediates Used in Paint, Varnish, Lacquer, and Related Products".
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Acknowledgements

The authors acknowledges the Ministry of Economy, Industry and Commerce, for the financial support of this study. Specifically, they wish to thank Dr. Vega-Baudrit for his support to this study, as well as providing LANOTEC's technical infrastructure.