Research Journal of Nanoscience and Engineering
Volume 3, Issue 1, 2019, PP 5-9
ISSN 2637-5591

Introduction and Characterization of Local CU Wire as A Reducing Media for CHN Analyzer

Ahmed Easa[1], Ahmed Abou Elezz[2], Ahmed Elkhatat[3], Saeed Al-Meer[1]*

[1]*Central Laboratory Unit, Qatar University, P. O. Box: 2713, Doha, Qatar*
[2]*Environmental Science Center (ESC), Qatar University, P.O. Box 2713, Doha, Qatar*
[3]*Department of Chemical Engineering, Qatar University, P. O. Box: 2713, Doha, Qatar*

***Corresponding Author:** Saeed Al-Meer, Central Laboratory Unit, Qatar University, P. O. Box: 2713, Doha, Qatar*

ABSTRACT

A new reaction tube was tested for CHN content in six different ranged standard materials by using a CHN analyzer based on the local copper wire as a reducing agent. The obtained results using the local copper wire were too close to the theoretical values with very low variability (<4%) between replicates and very high accuracy (98-101%). Scanning electron microscope (SEM), energy dispersive X-ray analysis (EDAX) and Raman analysis instruments were displayed a data close to the high pure standard electrolytic copper (manufacturer reaction tube). It is observed that the physical properties of a local Cu wire are identical with standard electrolytic copper. It concluded that local copper wire s has efficient and might be used as a reducing agent in CHN analysis.

Keywords: *Reaction tube; local copper wire; CHN content; reducing agent; standard materials*

INTRODUCTION

Carbon, Hydrogen and Nitrogen percentage are essential parameters, especially in studying the new compound and materials, which appears as a result of organic chemistry in many fields. There are several methods and devices have been developed widely in the last 20 years, the measurement depends on oxidation of the organic compound to some oxidase and reduces of these oxides, the reducing agent is varied from company-to-company and device to device. Copper is one of the essential reducing agents that is used in the elemental analyzer [1], there are many forms of copper, one of them called electrolytic copper, which is used in the elemental analyzer. The maker suggests using a high pure electrolytic copper in a wire form (figure 1). In the present work, another type of copper wire was purchased locally and used as a reducing agent, to reduce the cost of analysis. This type of wire usually used as a conducting agent in a truck battery.

Copper is one of the best both thermal and electrical conductor[2]. In the current world, the highest percentage of copper mined and refined is used to manufacture electrical appliances. It is also used in building construction, industrial machinery and more importantly in the recent studies copper as proven to be useful in the health sector in the treatment of diseases[3]. In organic reactions, copper is essential for reducing samples. Our study copper is one of the agents used in the elementalanalyzer[4]. Elemental analysis is defined as a process of determining the isotopic composition of substance like soil samples, water, mineral and other related chemical components.

Elemental analysis can be carried out in a variety of methods and devices. Chemical reactions entail combusting the sampled elements in excess oxygen, then tap the byproducts obtained from the reaction which include carbon dioxide, water, and nitric oxide. In this process, the electrolytic copper form is used with an instruction of using it in its highest pure form[5]. The Elemental analyzer can quantitatively determine the amount of carbon, hydrogen, nitrogen and sulfur present in an element using pure copper electrolyte. In this case study, the aim is to investigate the possibility of replacing the pure form of the electrolyte copper in the reaction tube with locally purchased copper cables, which are used as electrical conductors in truck batteries with the aim of reducing the elemental analysis cost.

Materials and Method

Qualitative morphology of the local copper wire was determined using scanning electron microscopy (SEM) model FEI Nova NanoSEM 450 Electron Nanoscopy Instrument (Figure 1, 2). A representative portion of the Copper wire sample sprinkled onto a double-sided carbon tape mounted on SEM stub at low-vacuum mode (110 Pa) was applied to test non-conductive samples without a need for deposit a conductive film to carry away an electric charge. Point microanalysis also done for selected local copper wire using the EDAX Bruker Nano X-flash 5010 analytical system. Raman spectroscopy analysis was performed with a Raman microscopy system (DXR Raman microscope, Thermo Fisher Scientific, USA). A He−Ne laser (λ = 633 nm,) was used as the excitation source. An epiillumination microscope (Olympus, Model BX-KMA-LED) with an objective (Olympus, 50X/0.75) was used to focus and collect the incident and scattered laser light during electrochemical measurements.

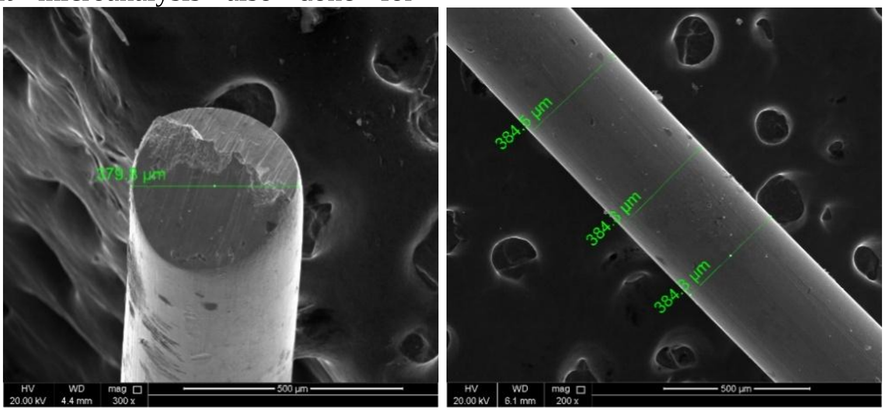

Figure1. *SEM image of high pure electrolytic copper suggested by the device manufacturer*

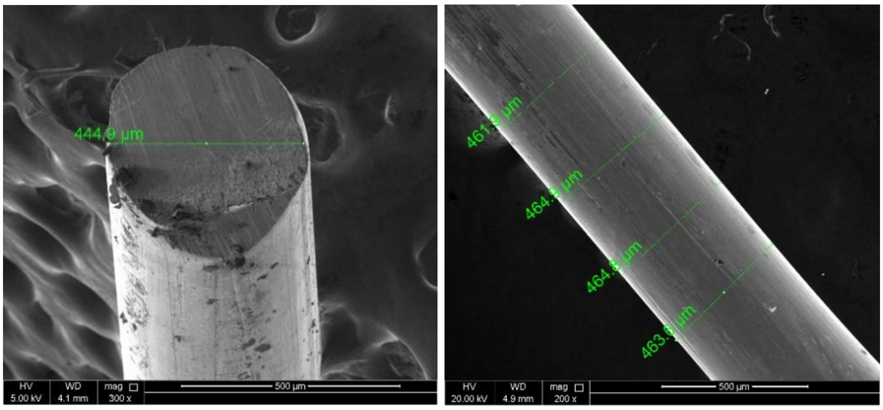

Figure2. *SEM image of Local copper wire*

Reaction Tube Preparation

About fourteen cm long of local copper wire was employed in the reaction tube of the CHN elemental analyzer (Flash 2000) as a reducing agent and followed by 5cm Copper oxide as an oxidizer.

Sample Preparation and Analysis

Approximately 2.5 ± 0.5 mg of each reference material or sample was weighted and capsuled in a thin capsule. The capsule was closed and introduced into a reaction tube. The sample was heated up to 900˚C for complete combustion; then the combustion product was passed through a copper oxide layer that assists to converting all CHN forms in the sample to the element oxides and water in the presence of oxygen gas[6]

Heliumgas carried oxides through the Cu wire (local copper) to reduce all oxide formsinto elements thatcarried into GC-column by Helium for separation at a particular retention time (70 – 270 sec). A thermal conductivity detector (TCD) was quantified CHN elements, and the result was recorded as a percentage. Figure 3 illustrates the stages of (CHN) analysis by using flash 2000 elemental analyzer.

The Efficiency of Local Copper Wire

Five reference materials (aspartic acid, BBOT, Acetanilide, Sulphanilamide, Soil std, and Benzoic acid) obtained from Thermos-Science were used to examine the efficacy of local copper wire to the CHN analysis. Five independent repetitions were tested for CHN in each reference material. Relative Standard deviation (RSD) and recovery percentage were calculated for each element compared with a theoretical value (Table 1). Soil std was also tested as a low range sample to assure efficiency of the used copper.

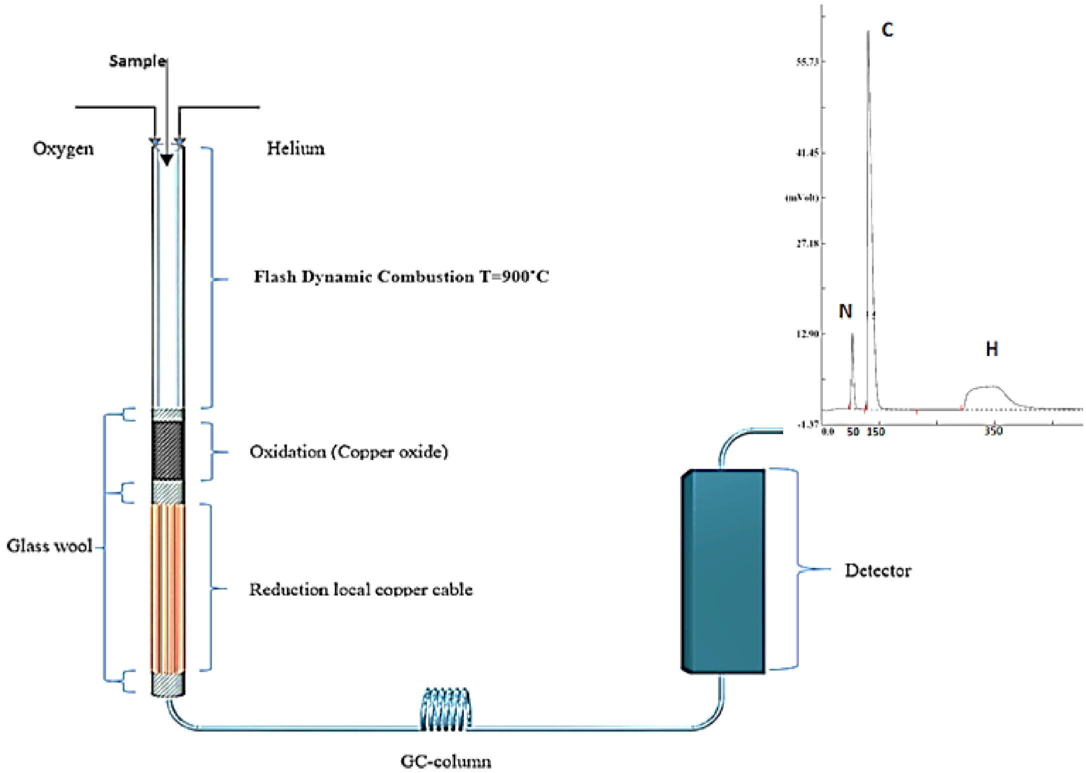

Figure3. *CHN analysis diagram including a reaction tube configuration with a local copper wire, GC-column separation and TCD*

Results and Discussion

Characterization of Local Copper Wire

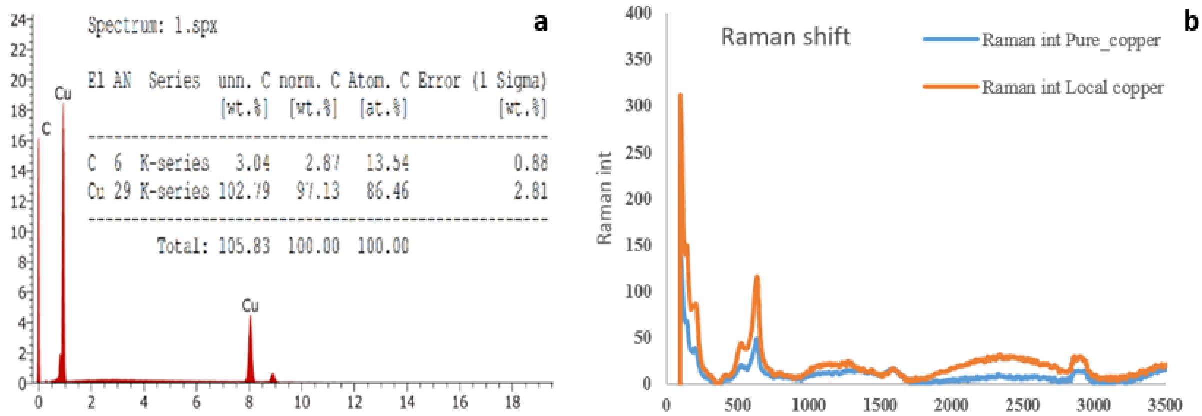

***Figure4**. (a) ADEX, (b) Raman results of local copper wire*

EDAX and Raman analysis of the local copper wire, Figure (4) shows that there was a very slight difference in performance in both pure copper electrolyte and the local copper. The effect of the local copper on the accuracy and precision of CHN results is indicated in Table (1) which reflects that the volumes of nitrogen carbon and hydrogen produced as a result of the redox reaction using the local wire are slightly varied between the elements. Figure 5 demonstrates the closeness between the mean analyzed concentrations of six different referencematerialsand itstheoretical values in each element with very low variation.

Table1. *Relative Standard Deviation (RSD) and the Recovery percentage (R%) of 6 CRMs replicates obtained from the CHN Analyzer with Local copper wire.*

Reference Material	CHN	N	Mean%	Std. Deviation	RSD	Theoretical Values%	Recovery%
Acetanilide	C	5	70.66	0.13	0.18%	71.09	99%
	H_2	5	6.73	0.06	0.81%	6.71	100%
	N_2	5	10.39	0.10	0.99%	10.36	100%
BBOT	C	5	72.27	0.29	0.40%	72.56	100%
	H_2	5	6.020	0.09	1.53%	6.11	99%
	N_2	5	6.46	0.09	1.33%	6.49	100%
Sulphanilamide	C	5	41.63	0.15	0.35%	41.85	99%
	H_2	5	4.57	0.04	0.81%	4.68	98%
	N_2	5	16.21	0.19	1.18%	16.26	100%
Soil std	C	5	2.18	0.03	1.51%	2.21	99%
	H_2	5	0.86	0.01	0.91%	0.86	101%
	N_2	5	0.20	0.01	3.92%	0.20	101%
Aspartic acid	C	5	35.99	0.10	0.28%	36.14	100%
	H_2	5	5.26	0.10	1.99%	5.31	99%
	N_2	5	10.34	0.05	0.49%	10.49	99%
Benzoic acid	C	5	68.75	0.08	0.12%	68.78	100%
	H_2	5	4.87	0.06	1.28%	4.91	99%

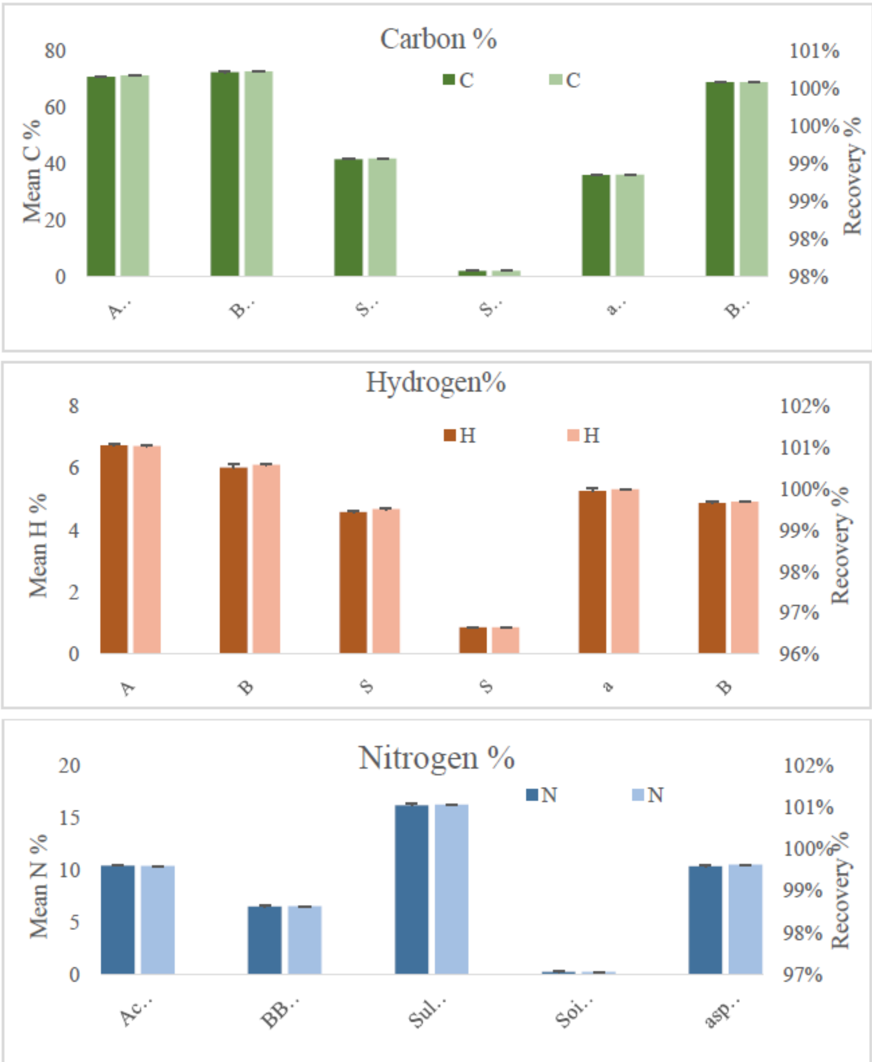

Figure5. *Comparison between the mean measured CHN values and the theoretical values of the reference materials*

Conclusion

Five different reference materials were tested for elemental analysis under constant conditions with the local Cu wire. The results indicated very low variability between the replicate concentrations (less than 5%) and very high accuracy (98- 101%) compared with the reference values, and regarding also to the significant price difference, that are qualifying the local wires to take the place of the manufactured pure copper electrolyte as a reducing agent.

References

[1] A. Pietro Reverberi, M. Salerno, S. Lauciello, and B. Fabiano, "Synthesis of copper nanoparticles in ethylene glycol by chemical reduction with vanadium (+2) salts," *Materials (Basel).*, vol. 9, no. 10, pp. 1–11, 2016.

[2] Carl D. Millholland, "Another Reason Copper Is One of the Most Important Metals - Analyzing Metals," 2016. [Online]. Available: https://www.thermofisher.com/blog/metals/another-reason-copper-is-one-of-the-most-important-metals/. [Accessed: 13-Jul-2018].

[3] C. D. Millholland, "Another Reason Copper Is One of the Most Important Metals," *Thermo Fisher Scientific*, 2014. [Online]. Available: https://www.thermofisher.com/blog/metals/another-reason-copper-is-one-of-the-most-important-metals/.

[4] S. Mathews, R. Kumar, and M. Solioz, "Copper reduction and contact killing of bacteria by iron surfaces," *Appl. Environ. Microbiol.*, vol. 81, no. 18, pp. 6399–6403, 2015.

[5] I. Baneschi, L. Dallai, G. Giazzi, M. Guidi, and L. Krotz, "A method for the definition of the carbon oxidation number in the gases dissolved in waters and the redox variations using an elemental analyser (FlashEA 1112). Preliminary data from a stratified lake," *J. Geochemical Explor.*, vol. 124, pp. 14–21, 2013.

[6] V. P. Fadeeva, V. D. Tikhova, and O. N. Nikulicheva, "Elemental analysis of organic compounds with the use of automated CHNS analyzers," *J. Anal. Chem.*, vol. 63, no. 11, pp. 1094–1106, 2008.

Citation: *Saeed Al-Meer, et al. "Introduction and Characterization of Local CU Wire as A Reducing Media for CHN Analyzer", Research Journal of Nanoscience and Engineering, vol. 3, no. 1, pp. 5-9, 2019.*