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Spectrophotometric fast determination of Cu and Zn in Steel Cord Brass Coating via an Effective Masking Approach

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ABSTRACT

The Cu^{2+} and Zn^{2+} ions were determined in the steel cord brass coatings with high precise. HNO_3 65% was used for digesting the coatings and releasing the ions into environment. The outlier test, normality test, F-test and T-test were used for data analysis. As a fast, precise and simple analysis method, it can be utilized in industrial laboratories. Moreover, quantometery method can be replaced by this type of analysis, which demonstrates its importance from the industrial viewpoint as an economical pathway. Using accessible chemicals a laboratory instruments is another benefits of our proposed method.

1. Introduction

With the development of the automotive industry, an increment in request for more progressive tires is considered. Nowadays, the rubber mixes, textile fibers and steel wires as a complex mixture are joined together in the vulcanization process [1-3]. The highest effect on the life content of tires, utilized in all vehicle types is demonstrated via the tire's internal structure and the amplification used. Either steel wire or its derivatives as ropes or strands are utilized, named the steel cord, for reinforcing tires [4-17], which, imprinted in an elastomer, are asked to make the structure harder, while imparting to it the suitable. The service characteristics of steel cord are considerably affected by its chemical composition, wire drawing and cord winding processes. The used wires for steel cord production, made of unalloyed pearlitic steel, involving from 0.70 to 0.95% of carbon [18-22].

In the continuation of my previous study about chemical analysis of steel cord [23] and due to the fact that the chemical characteristics and properties of the steel cord have a remarkable and decisive effect on tire behavior in the various environmental conditions, the chemical analysis of steel cord seems very useful and necessary.

2. Experimental

0.35 grams of unbent steel wire was weighed and transferred to a 100 ml beaker. A few amount of a 50-50 solution of chloroform-toluene were added to the beaker and washed, completely. The excess solution was discarded and the samples were transferred to an oven with a temperature of 105 °C to dry. After drying, the samples were transferred to a desiccator to cool and transferred to a 200 ml flask. 5 ml of nitric acid (65%) was added, the cap of the flask was placed and it was vigorously stirred for 1 minute. Using a magnet, the sample was removed and washed again with 5 ml of nitric acid in a balloon tube. The samples were washed with deionized water and the samples were removed from the flask and the flask was filled to volume. A magnet is placed inside the balloon to make it completely homogeneous.

To analyze the copper ion, a 10% solution was prepared from the prepared solution and the copper ion test was performed using a spectrophotometer in the 1695 method: 10 ml of the prepared solution was transferred to one cell as a blank and in the second cell 10 ml of the solution was added along with the copper reagent. After 3 minutes of effective stirring, the solution was placed in the device and the result was recorded.

In order to analyze zinc ions, 20 ml of the prepared solution was transferred to a 100 beaker. One drop of triethanolamine solution, 3 drops of buffer solution (including potassium cyanide, borax, and sodium hydroxide) and one package of zincone reagent were added to it. At this stage, the color of the solution turned

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orange. 10 ml of this solution was transferred to a cell and tested by method 3850 spectrophotometer. Another 10 ml of the solution was added to the second cell and 0.5 ml of cyclohexanone was added to it. Only 1 drop of ethanolic DPPH solution was added to it and the solution was vigorously shaken. The timer of the device was turned on and after 3 minutes the solution was allowed to rest and then it was placed in the spectrophotometer and the result was recorded.
The results were obtained according to the following equations:

$\%Cu = C_{Cu2+(ppm)} / C_{Cu2+(ppm)} + C_{Zn2+(ppm)}$

$\%Zn = C_{Zn2+(ppm)} / C_{Cu2+(ppm)} + C_{Zn2+(ppm)}$

3. Results and discussion

In this research, outlier test (Fig. 1), normality test (Fig. 2), F-test (Fig. 3) and t-test (Fig. 4) were used for data analysis. In the outlier test, if P value ≥ 0.05, its means that there is not an outlier data. In the normality test, if the P value ≥ 0.05 it means that the data distribution is normal and is carried-out with an acceptable tolerances. In F test, the P value ≥ 0 means that the data averages are in the acceptable range. Finally, in the T test if the P value ≥ 0, it means that data middle is in the acceptable range.

Table 1. Cu^{2+} and Zn^{2+} percentages obtained by spectrophotometer.

	C1	C2
	CU	
1	A	64.09
2	A	60.89
3	A	60.90
4	A	63.16
5	B	63.70
6	B	61.46
7	B	63.28
8	B	64.90

	C1	C2
	Cu 2	
1	A	64.91
2	A	61.19
3	A	63.71
4	A	63.97
5	B	64.74
6	B	65.93
7	B	64.49
8	B	60.91

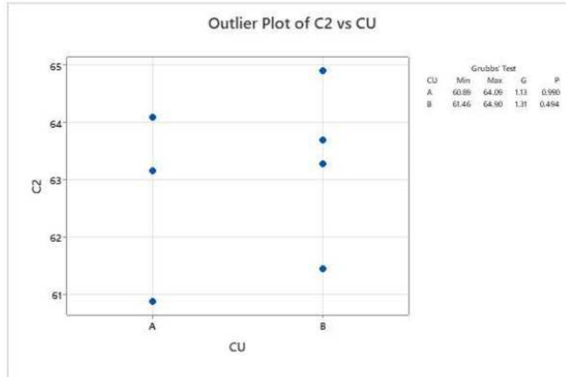
	C1	C2
	Zn 1	
1	A	35.91
2	A	36.84
3	A	39.11
4	A	39.10
5	B	36.30
6	B	38.54
7	B	36.71
8	B	35.10

	C1	C2
	Zn 2	
1	A	35.09
2	A	38.81
3	A	36.29
4	A	36.03
5	B	35.26
6	B	34.07
7	B	35.51
8	B	39.09

Grubbs' Test

Variable	CU	N	Mean	StDev	Min	Max	G	P
C2	A	4	62.260	1.621	60.890	64.090	1.13	0.990
	B	4	63.335	1.426	61.460	64.900	1.31	0.494

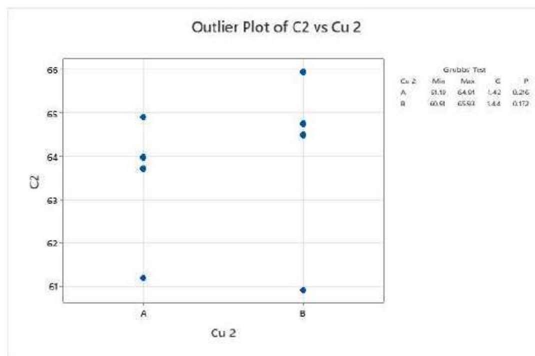
* NOTE * No outlier at the 5% level of significance



Grubbs' Test

Variable	Cu 2	N	Mean	StDev	Min	Max	G	P
C2	A	4	63.445	1.589	61.190	64.910	1.42	0.216
	B	4	64.02	2.16	60.91	65.93	1.44	0.172

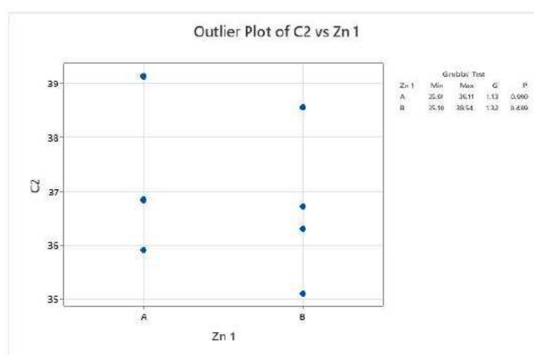
* NOTE * No outlier at the 5% level of significance



Grubbs' Test

Variable	Zn 1	N	Mean	StDev	Min	Max	G	P
C2	A	4	37.740	1.621	35.910	39.110	1.13	0.990
	B	4	36.663	1.426	35.100	38.540	1.32	0.489

* NOTE * No outlier at the 5% level of significance



Grubbs' Test

Variable	Zn 2	N	Mean	StDev	Min	Max	G	P
C2	A	4	36.555	1.589	35.090	38.810	1.42	0.216
	B	4	35.98	2.16	34.07	39.09	1.44	0.172

* NOTE * No outlier at the 5% level of significance

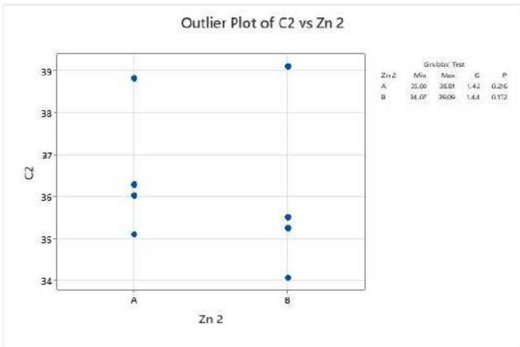
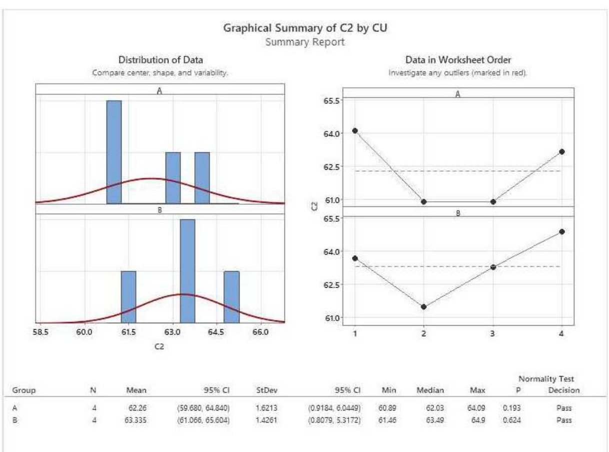


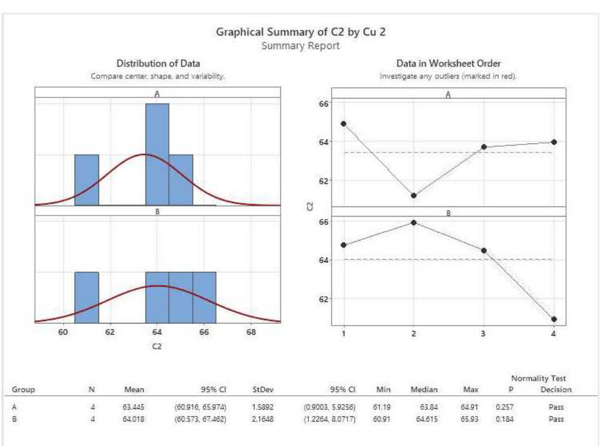
Fig. 1. Outlier test analysis for Cu^{2+} and Zn^{2+} ions.

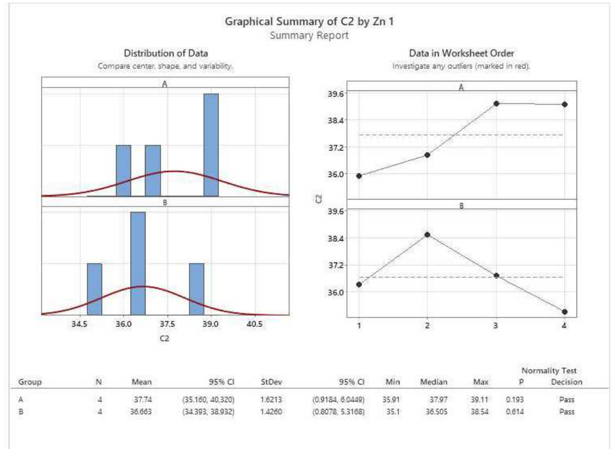
To perform the Grubb's test, G_{exp} is calculated with the following equation.

$G_{exp} = |Xq - X| / S$

This parameter is calculated using Minitab software in this article. Of course, it should be said that it could be calculated with Excel and SPSS software, which is based on the average data and the standard deviation of outlier data.







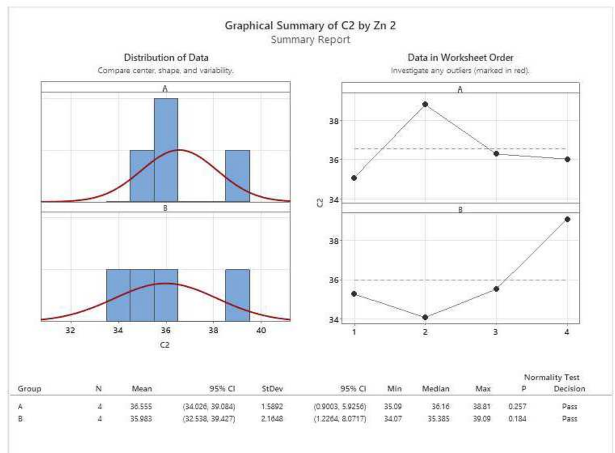


Fig. 2. Normality test analysis for Cu^{2+} and Zn^{2+} ions.

The data normality test is a method to determine whether the distribution of the collected data has a normal or normal distribution. The presuppositions of normality, linearity, and the sameness of the data distribution are that by creating a probability diagram of normality (in the shape of a bell and also symmetrical to the mean), it tests the assumption whether the data follows a normal distribution or not. This distribution has the following features:

1- Being symmetrical; The maximum height is in the middle. Half of the scores are above the average and the other half are below the average.

2- The values of median and mean are equal.

3- The distribution curve is like a bell.

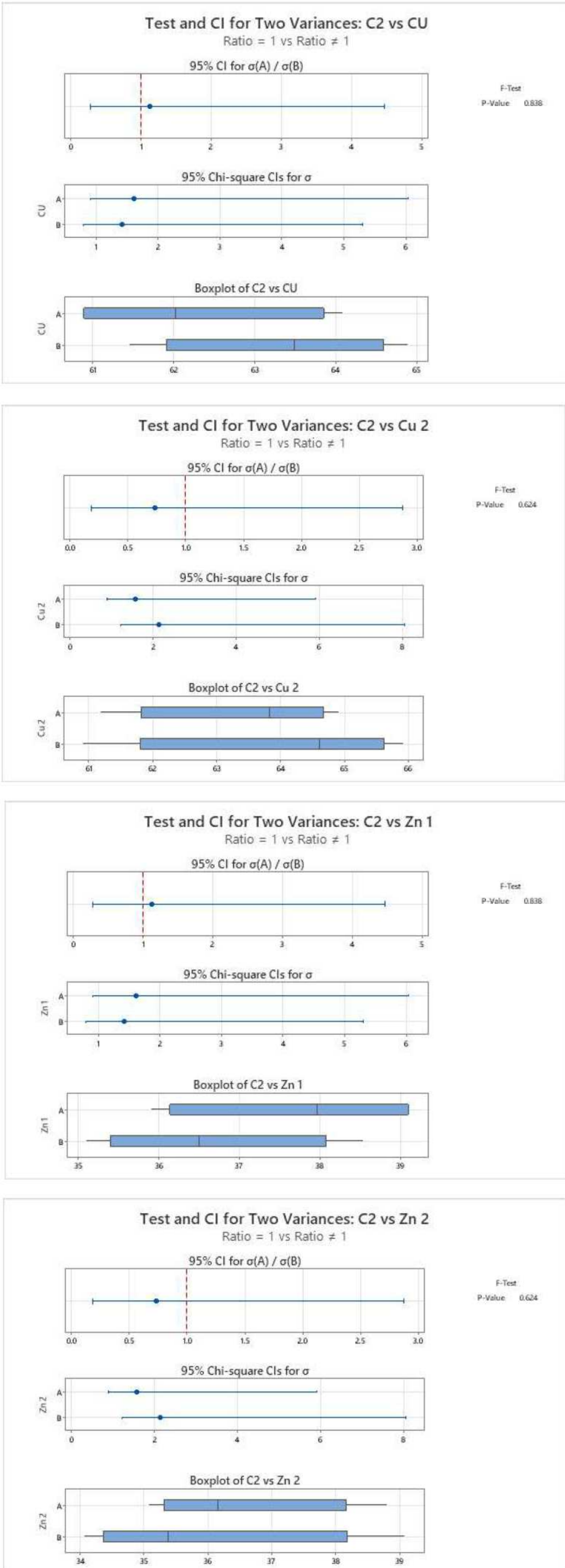


Fig. 3. F test analysis for Cu^{2+} and Zn^{2+} ions.

The F test or Ronald-Fisher test provides the possibility to compare the variance of two different data sets to determine a statistically significant difference. To calculate experimental F (F_{exp}) and compare it with critical F (F_{crit}), F test is performed. If $F_{exp}>F_{crit}$, the variances of the two data sets used are statistically different. To obtain F_{exp}, the following relationship is used:

$$F_{exp} = S_1^2/S_2^2$$

F_{exp} is determined by the ratio of the variance of the square of the standard deviation.

Descriptive Statistics: C2

CU	N	Mean	StDev	SE Mean
A	4	62.26	1.62	0.81
B	4	63.34	1.43	0.71

Estimation for Difference

Difference	Pooled StDev	95% CI for Difference
-1.08	1.53	(-3.72, 1.57)

Test

Null hypothesis H_0: $\mu_1 - \mu_2 = 0$
Alternative hypothesis H_1: $\mu_1 - \mu_2 \neq 0$

T-Value	DF	P-Value
-1.00	6	0.358

Descriptive Statistics: C2

Cu 2	N	Mean	StDev	SE Mean
A	4	63.45	1.59	0.79
B	4	64.02	2.16	1.1

Estimation for Difference

Difference	Pooled StDev	95% CI for Difference
-0.57	1.90	(-3.86, 2.71)

Test

Null hypothesis H_0: $\mu_1 - \mu_2 = 0$
Alternative hypothesis H_1: $\mu_1 - \mu_2 \neq 0$

T-Value	DF	P-Value
-0.43	6	0.685

Descriptive Statistics: C2

Zn 1	N	Mean	StDev	SE Mean
A	4	37.74	1.62	0.81
B	4	36.66	1.43	0.71

Estimation for Difference

Difference	Pooled StDev	95% CI for Difference
1.08	1.53	(-1.56, 3.72)

Test

Null hypothesis $H_0: \mu_1 - \mu_2 = 0$
Alternative hypothesis $H_1: \mu_1 - \mu_2 \neq 0$

T-Value	DF	P-Value
1.00	6	0.357

Descriptive Statistics: C2

Zn 2	N	Mean	StDev	SE Mean
A	4	36.55	1.59	0.79
B	4	35.98	2.16	1.1

Estimation for Difference

Difference	Pooled StDev	95% CI for Difference
0.57	1.90	(-2.71, 3.86)

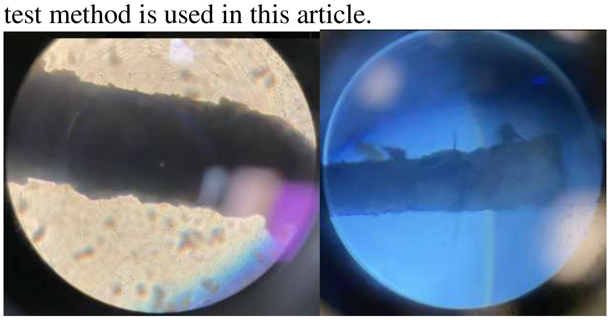
Test

Null hypothesis $H_0: \mu_1 - \mu_2 = 0$
Alternative hypothesis $H_1: \mu_1 - \mu_2 \neq 0$

T-Value	DF	P-Value
0.43	6	0.685

Fig. 4. T test analysis for Cu^{2+} and Zn^{2+} ions.

The average results are compared using the T test method. In the data community of equal variances, averages are compared with this method. Two sample T-test method is used in this article.



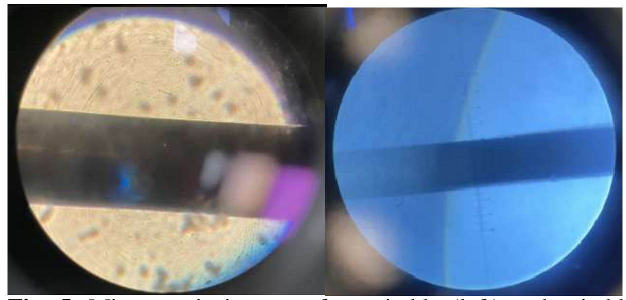


Fig. 5. Microscopic images of unsuitable (left) and suitable (right) interactions of HNO_3 with sample.

Conclusion

In this method, copper and zinc ions were released from the interaction of nitric acid with steel wire pieces. After interacting with nitric acid and releasing the ions in the solution, the amounts of the mentioned ions were measured with a spectrophotometer with high accuracy. This method can be carried-out instead of the time-consuming and expensive current methods in related industries. The high precise and accuracy of the obtained results as well as its usefulness guaranties its efficiency in the industrial laboratories.

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