



## **Novel Non-parametric Assessment of Heavy Metals in Coated Card Wastes**

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### **Authors' contributions**

*This work was carried out in collaboration between all authors. Authors CIAN and AO designed the study, wrote the protocol and managed the analyses of the study. Authors AUN and PCN managed the literature searches, performed the statistical analysis, wrote the first draft of the manuscript. Author AUN produced the final draft. All authors read and approved the final manuscript.*

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### **ABSTRACT**

Non-parametric tests were applied for the first time to coated mobile card waste in order to determine the correlation within risk assessment and instrumentation. The AAS concentrations of all the metals were higher than their XRF concentrations except in Al and Ag metals. Cr had the highest distribution pattern while Ag metal had the lowest respectively. The target hazard quotient (THQ) for all metals were below minor hazard level. On the contrary, only Fe and Zn were below the Oral reference dose (RfD) value and the daily metal intake (DMI) respectively. The Wilcoxon-Mann-Whitney test showed no significant difference between the concentration of the metals in XRF and AAS analysis results but identified the unusual behavior of Ag and Al metals; hence it depicted that both AAS and XRF analysis results were not sensitive to detect the different metal concentrations in the cards. Hence, similar coating specifications are utilized. The Kruskal Wallis test and Spearman correlation coefficient showed that the results from AAS and XRF were significantly different. Hence it showed that instrumentation showed sensitivity of metal concentrations from the recharge cards. This correlated with THQ, DMI, health risk index (HRI) risk

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assessment parameters that different coating specifications were used causing different values at same sampling points. Therefore, the results established that non-parametric tests are good analytical tools.

*Keywords: Non-parametric; card-waste; heavy-metals; risk-assessment.*

## 1. INTRODUCTION

The Mobile phone industry in Nigeria is a multi-billion-naira industry with vast opportunities for introduction of product varieties and consumer choices. However, this technology has brought certain socio-economic downside such as indebtedness of phone owners to service providers, the challenges of network failure, unreliable power supply for charging phone batteries, stealing of mobile phones, and untrained repair technicians handling smart phones [1]. On the other hand, the scientific challenge of this technology that has permeated the Nigerian market is the significant increase of waste and other associated accessories emanating from use of mobile phones. Such accessories include the traditional coated recharge cards popularly known as scratch cards. This recharge cards are metal coated and has been identified to contain heavy metals such as Cu, Ag, Cr, Ni, Cd, Al and Zn at significant levels [2,3]. They are often disposed on the roadsides or in mixed domestic waste bags which eventually are disposed in dumpsites, landfills or open burning as a municipal solid waste [4]. The soils and ground water when contaminated are capable of causing somatic mutations and oxidative stress in living cells [5] and adversely affects soil flora and fauna [6]. There have been recommendations for e-recharge portals (systems) to eliminate this problem. However, frequent network subscriber complaints of debiting of bank cards without crediting their phone SIM cards by network providers creates apprehension to the existing and intending user, hence the continued patronage of metal coated recharge cards [7].

In addition, humans have remained at higher risk of occupational exposure to heavy metals found in the coatings of recharge cards. They metal such as Fe, Ni, Cr, K, Ca, Sc, Ti, Zn and Cu, and Mn have all been detected and poses health threats to man [8]. For example, speciation study of heavy metals in recharge card coatings have determined Ti as significantly present in undesirable concentrations and Mn element as the most mobile among other elements [9]. Thus mobile phone wastes (including recharge cards)

have been classified as a hazard without the consumer been aware of the dangers associated with its usage [10,11]. These dangers have been predominant in academic institutions which were described as been persistent and prevalent, and thus their hazard reduction should be supported at all levels [12,9].

Hence we made a conscientious search for non-parametric predictive evaluation of data on heavy metals risk, exposure and distribution in coated card waste but found a knowledge gap. To this end therefore, there is need to carryout risk assessment of the chemical content of mobile phone recharge cards sold in a localized academic environment, and to evaluate the data using non-parametric data analysis for predictive behavior in their distribution, exposure and risks.

## 2. MATERIALS AND METHODS

### 2.1 Sample Preparation

Two brands of mobile coated recharge cards from network A and B were selected for the experiment. A total number of 25 samples were obtained from each network. The samples of the network coated cards selected for analysis were purchased from four different points in Federal University of Technology Owerri, Nigeria. The samples were scratched into a clean cellophane bag using a plastic scrapper and kept in dry place. Fresh standard solutions were prepared for the external calibrations of XRF and AAS spectroscopic analysis using nitrate salts of all metals. The samples were run in triplicates and average values of the metal concentrations from the recharge coatings were reported.

### 2.2 AAS Analysis

The method described by previous studies [3,8] was modified to achieve complete and faster digestion. 0.05 g of sample was weighed into a 250 ml conical flask for digestion. 10 ml of Perchloric acid, nitric acid and sulphuric acid were added into the mixture in the ratio of 1:2:2 and heated on a hot plate in a fume chamber. The mixture was heated at 110°C for half an hour until white fumes appeared and sample

slowly evaporated to dryness. The samples were filtered through Whatman's No. 1 filter paper and made up to 50 ml with distilled water. The clear solution was then analyzed using AAS. Sample recovery was up to 93% indicating good replicability.

### 2.3 XRF Analysis

The metal quantification was carried out using an XRF spectrometer. 1.0 g of sample was worked into a pellet shape using a hydraulic press of 13 mm diameter Spec-Cap. A foil was used to hold it in place while the x-ray-beam was initiated to irradiate the sample for 1000 seconds which produced the X-ray fluorescence peaks. The peaks of elements of interest were identified and concentrations determined using same XRF quantification software [9].

### 2.4 Risk Assessment

The distribution of heavy metals in the recharge cards were studied using the coefficient of Variation (CV) to show the distribution patterns of the heavy metals in the scratch cards. The CV was calculated using the formula below [13].

$$CV = \left( \frac{STDEV}{Mean} \right) \times 100 \quad (1)$$

where; STDEV is the Standard deviation.

The exposure rate was determined using the target hazard quotient (THQ) [14]. It was used to estimate the probability of the population that has adverse effect from exposure of the hazard. Hence it provides risk level as a result of pollutant exposure and the formulae is given below

$$THQ = \frac{Ef \times ED \times CR \times Mc \times 10^{-3}}{RfD \times BW \times AET} \quad (2)$$

where Ef is the exposure frequency of 365 days/year; ED is the exposure duration of 70 days; CR is the consumption rate of 0.01 g/day for average adult (been the average amount retained by nails when scratched with fingernails); Mc is the metal concentration in recharge cards in mg/kg; RfD is the reference oral dose by ATSD index; BW is the body weight assumed to be 65 kg for average adult; while AET is the average exposure time which is 365 days/year  $\times$  ED; and  $10^{-3}$  is the conversion unit factor. If the HQ value is  $>1$  then it indicates the state of risks to the environment. If:  $HQ < 0.1$ ;

hazard exists; HQ 0.1-1.0: hazard is low; HQ 1.1-10: hazard is moderate; HQ  $> 10$ : hazard is high.

The health risk assessment was determined using the daily intake of metals (DIM); the formula is given below [8].

$$DIM = \frac{C_{metal} \times D_{intake}}{B_{average}} \quad (3)$$

Where DIM = Daily intake of metals  
 $C_{Metal}$  = Heavy metal concentration in coatings;  
 $D_{Intake}$  = Intake of metals (about 10% of what was scratched and retained under the finger nails);  
 $B_{average}$  = Average body mass of an adult (65 kg)

For the DIM all the calculated values obtained will be compared with the standard values given for the oral reference doses (RfD). Additionally, the health risk index (HRI) will be determined using Daily Intake of Metals in food (DIM) and Oral Reference Dose (RfD) through the following formula below [8].

$$HRI = DIM/RfD \quad (4)$$

If the value of HRI is less than 1 ( $HRI < 1$ ), then it means that the health risk exposed to the population is acceptable.

The metal concentrations from AAS/XRF analysis results would be evaluated with THQ, DMI, HRI and RfD since there are currently no regulation/specifications for production of coated recharge cards in Nigeria. Moreover, similar work has reported increasing usage among the young (scratched and retained in fingernails) and need for regulations [3,5,9].

### 2.5 Non-Parametric Assessment

The Wilcoxon-Mann-Whitney test is a non-parametric type of test homologous to the t-test. It thus can be used in place of t-test for dependent variables and operates without condition of normality of data [15]. It can be used when there is no assumption that the dependent variable is normally distributed and the variable is also assumed to be at least ordinal. The formula for calculating Wilcoxon-Mann-Whitney test is given below.

$$U = N1 \times N2 + \left[ \left( N1 \times \frac{N1 + 1}{2} \right) - R1 \right] \quad (5)$$

$$\mu u = \frac{N1 \times N2}{2} \quad (6)$$

$$\sigma\mu = \sqrt{\frac{N1N2(N1 + N2 + 1)}{12}} \quad (7)$$

$$Zscore = \frac{(U - \mu u)}{\sigma\mu} \quad (8)$$

Where N1 is count of samples in 1st group

N2 is count of samples in 2nd group

U is the U statistics

R is the sum of ranks

$\sigma u$  is the standard deviation

$\mu u$  is the calculated mean

Z score is the z value to be compared with critical value

The Kruskal Wallis test is a non-parametric test used when there is one independent variable with two or more levels and an ordinal dependent variable [15]. Sometimes it is referred as the non-parametric version of ANOVA. Moreover, when the assumptions of ANOVA that population variance are equal or that populations are normally distributed are not met, then non-parametric (Kruskal Wallis test) is used. The Kruskal Wallis test is determined through the following formula given

$$H statistics = \frac{12}{N(N + 1)} \left( \frac{R1^2}{n1} + \frac{R2^2}{n2} + \frac{Rk^2}{nk} \right) - 3(N + 1) \quad (9)$$

Where n1 is number of samples in 1st group

n2 is number of samples in 2nd group

nk is number of samples in kth group

N is the total number of samples

k is the number of samples

df is the degrees of freedom = k-1

The Spearman rank correlation coefficient is a non-parametric tool used when the variables are assumed to be normally distributed and having intervals or if the assumption for testing hypothesis in Pearson's coefficient (p=0) is not met, then non-parametric equivalent (spearman correlation) is used. The values obtained from the variables are converted into ranks and then correlated accordingly [15]. The spearman rank would be calculated using the following formula

$$r_s = 1 - \frac{6\sum d^2}{n(n^2 - 1)} \quad (10)$$

d = difference in ranks

n = number of data pairs

$r_s$  = spearman rank coefficient

### 3. RESULTS AND DISCUSSION

#### 3.1 Sampling Analyses

The total metal concentrations obtained from AAS and XRF analysis results from each of the analyzed coated recharge cards were plotted against their four sampling points as shown in Fig. 1. The Vertical column depicts the concentration of the metals while the horizontal row shows the four respective sampling points. The first two bars for each metal shows their AAS concentrations while the last two showed their XRF concentrations for the same metal analyzed. A general observation revealed that AAS concentrations were higher than XRF concentrations in all metals except in Aluminum (Al) but also were significantly close in silver (Ag) metal concentrations. The metal concentrations at the four sampling points were generally in this trend, Al: 3 > 4>1>2; Ag: 1>4>3>2; Cr: 1>2>3>4; Cu: 1>2>4>3; Fe: 1>2>3>4; Zn: 1>2>3>4. Thus it would be concluded that sampling point 1 showed the highest concentration of metals followed by sampling point 2 as obtained from AAS, then sampling point 3 followed by sampling point 4 from XRF respectively. This may indicate the sensitivity of AAS spectroscopic analysis over X-ray Fluorescence in detecting metals such as Cr, Cu, Fe, Zn from recharge cards except in Al and Ag that showed significant deviation from the norm. On the overall, the metals showed highest average concentrations in this order Ag>Fe>Cr>Zn>Cu>Al.

This average concentration trend (Ag>Fe>Cr>Zn>Cu>Al.) confirms the presence of these metals in the card coatings. Their result was similarly confirmed by several researchers on the high concentrations of Fe and low concentration of Cu [8,9] in recharge card waste. However, the contrary trend of low Ag concentration and high Cu concentration has also been observed by another author [3]. Interestingly, the disposal of the card waste will eventually enrich the soil by all metals and affect the biota [5,16,17]. Hence the sorting of waste into different components to enhance disposal and reduce metals leaching into the soil and water bodies from waste dumpsites [4].

From the AAS analysis results, the average concentrations of the metals in Network A showed higher levels of metals present in their recharge cards than network B and had a trend as follows: Ag>Fe>Cr>Zn>Cu>Al at 4.87, 1.98, 1.62, 1.43, 1.05, 0.17 (mg/kg) respectively.

Similarly, the XRF analysis results of Fe, Cr, Zn and Al metals obtained from network A was higher than those of network B except for Ag and Cu metals at 4.02 and 0.19 (mg/kg) respectively.

### 3.2 Risk Assessment

The average concentration of the metals: Cu, Cr, Fe, Ag, Zn and Al from Atomic Absorption and X-ray fluorescence results as determined, were used to calculate their respective CV, HRI, THQ

and DMI values. The coefficient of variation (CV) can be seen on the upper left hand side of Fig. 2. The CV was applied to show the distribution patterns of the metals analyzed in the four different sites. Research has confirmed that higher CV values depicts greater uneven distribution of the metals and parameters in consideration. Thus from the Fig. 2 it would be observed that Chromium (Cr) had the highest CV at sampling points 2 and 4 respectively, followed by aluminum at sampling points 4, and 3 respectively. The lowest was silver at points 3

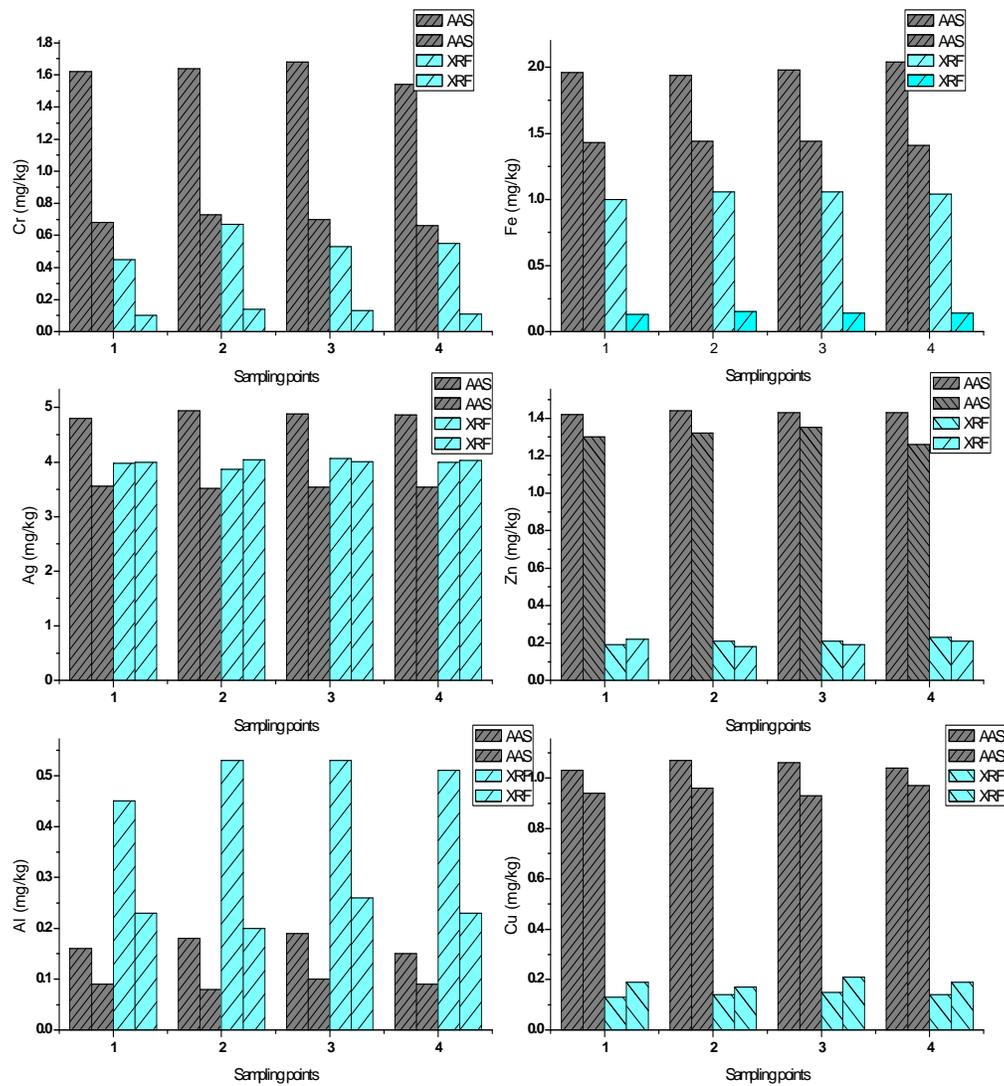


Fig. 1. Plot of AAS and XRF metal concentrations at the four sampling points: (From LHS-RHS; Cr, Fe, Ag, Zn, Al, Cu)

and 1 respectively followed closely by Iron (Fe) metal at sampling points 3 and 1 respectively. These demonstrated the absence of regulation of this card waste due to their great variation (Highest Cr: 16, Al: 10, Cu: 8, Zn: 8, Fe: 6, Ag: 2; Lowest Cr: 4, Al: 2, Cu: 2, Zn: 1; Fe: 2, Ag: 0.5) hence we suggest uneven distribution of the metals especially Cr and Al, Zn and Cu. Similarly, researchers have also observed higher coefficient of variation for Zn, Fe and Cr, Cu, a pointer to high exchangeable fractions of these metals that exist at the study sites [9,13].

The Target hazard quotient (THQ) is seen at the top right hand side of Fig. 2 above. The THQ is generally used to indicate the risk level that is associated with the metals exposure on both

average and high level risk. The THQ for all sampling sites were lower than 1 which depicts the absence of any major risk posed by the analyzed metals to users of recharge cards within the sampling site. On the other hand, the THQ for silver (Ag) ranged between 0.1 to 0.16 which confirmed that low hazard level of Ag metal hazard exists within the area. The other metals were below 0.1, an indication of minor health hazard and Zn was at micron levels. The THQ trend was in the following order Ag>Cr>Al>Fe>Cu>Zn. Thus the bioaccumulation of this metals through ingestion, inhalation and dermal contact over the years may eventually cause electrolyte imbalance, kidney and lungs defects and posed greater tendency to be carcinogenic at higher concentration [16,18].

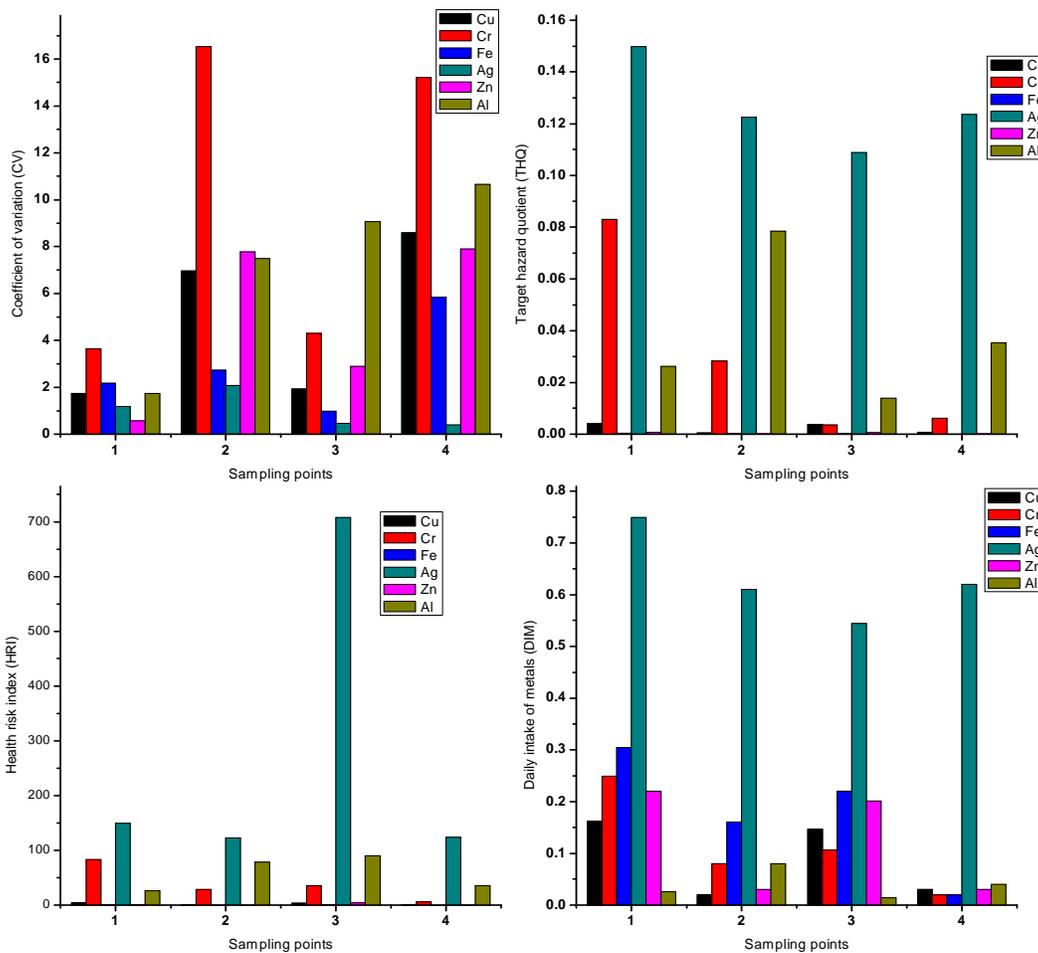


Fig. 2. Calculated risk assessment profile for metals. (From LHS-RHS; Coefficient of variation (CV), Target hazard quotient (THQ), Health risk index (HRI), Daily intake of metals (DMI))

The Daily metal intake was compared with that of the oral reference dose values from ASTM (ASTM, 2017) [19]. The results from Fig. 2 can be summarized at the Table 1.

The table clearly revealed that Ag, Al, Cr and Cu exceeded their standard reference values while the value of Fe and Zn were below the reference value. This revealed that minimum of 0.75 mg/kg is the daily intake of Ag for an average adult which is higher than standard RfD and the lowest consumed metal was Zn at 0.22 mg/kg and below the standard RfD. The common trend also observed, was that sampling point 1 was the significant point of maximum concentration levels. Hence, there is the likelihood that the retailer at sampling point 1 operates with different supplier, hence the higher likelihood of end users been exposed significantly more to metals than at other sampling points. Also similar studies have detected these metals at different concentrations and DIM levels, thus these metals exist as a fraction of what can be accumulated (ingestion, digestion or dermal contact) daily in the human body and may be excreted or retained in the human body tissues to induce adverse health effects [8,9].

The health risk index (HRI) was calculated and plotted as shown in Fig. 2 on the lower LHS. The tabular form is transcribed below in Table 2. The HRI values for all metals ranged from 708 (Ag) at sampling point 3 to 0.003 (Fe) at sampling point 4.

Thus except for Fe, (Zn and Cu at sampling point 4 and 2 respectively) the HRI of other metals were greater than 1 which implies that the health risk exposed to the population by use of recharge card users are unaccepted. this result however confirmed a correlation with DIM values above that showed silver (Ag) as the most ingested metals on daily exposure and followed by Al, Cr, and Cu in similar trend. However, the exceptions of Fe and Zn was still retained. Hence this result

similarly suggests unregulated and discriminatory use of Ag, Al, Cr and CU coating materials by the recharge cards producers which caused the variations observed in this research work. These values were also observed to show variation in similar work done by researchers. They also confirmed that several producers use different specifications for their card coatings and sold in different parts of the country to the consumers [8, 9].

### 3.3 Non-Parametric Assessment

Wilcoxon-Mann-Whitney test: The mean for each metal was determined and used to calculate the Wilcoxon-Mann-Whitney test. The Table 3 shows the constants and parameters obtained from the calculation. From the table it would be seen that the number of sample counts, standard deviation and calculated mean were the same while the sum of sample ranks differs. Accordingly, both the of XRF and AAS spectroscopic analysis results were rejected because the Zscore was greater than the critical value in both instrumental sampling. Thus there was no significant difference between the concentration of the metals analyzed by XRF and AAS. Hence we primarily suggest that both AAS and XRF were not sensitive to selectively detect the different metal concentrations in the cards [15].

Alternatively, either the same producer was supplying the two networks their recharge cards or that different suppliers were mainly using similar coating preparations and specifications. The result disagrees with HRI and RfD, CV and THQ that showed high variation among the calculated values as can be seen in Fig. 2. However, the Wilcoxon Mann-Whitney test consistently showed sensitivity to critically detect the similarity in concentration levels of Ag metal in both of XRF and AAS spectroscopic analysis as seen in Fig. 1. Hence, there was no significant difference in silver metal concentrations analysed by both instruments.

**Table 1. Maximum calculated RfD values against reference RfD values from the sampling sites**

Metals/RfD	Ag	Al	Cr	Cu	Fe	Zn
RfD (mg/kg)	0.005	0.001	0.003	0.040	0.700	0.300
Max Calc	0.75	0.05	0.25	0.15	0.30	0.22
Sampling point	1	2	1	1	1	1

**Table 2. Maximum and minimum calculated HRI values from the sampling points**

Metals	Ag		Al		Cr		Cu		Fe		Zn	
Max and Min	708	123	90	26	83	6	4	0.5	0.4	0.003	4	0.102
Sampling points	3	4	3	1	1,	4	1	2	1	4	3	4

**Table 3. Wilcoxon constants for AAS and XRF**

Wilcoxon test constants	N1	N2	R1	R2	U	Uu	$\sigma_u$	Zscore	Critical value	Hypothesis	Decision
AAS	6	6	45	33	12	18	6.245	-0.961	-1.96	-0.9607 > -1.96	Rejected
XRF	6	6	47	32	10	18	6.245	-1.28	-1.96	-1.281 > -1.96	Rejected

**Table 4. Kruskal Wallis constants of AAS and XRF**

Kruskal-Wallis constants	N1	N2	R1	R2	n	K	df	H statistics	H critical	Hypothesis	Decision
AAS	6	6	45	33	12	2	1	0.921	3.841	0.921 < 3.841	Accepted
XRF	6	6	47	31	12	2	1	1.394	3.841	1.394 < 3.841	Accepted

**Table 5. Spearman rank correlation coefficient and constants for AAS and XRF**

Spearman correlation constants	$d^2$	n	Critical value	$r_s$	Hypothesis	Decision
AAS	15	6	0.738	0.571	0.571 < 0.738	Accepted
XRF	30	6	0.738	0.143	0.143 < 0.738	Accepted

The Kruskal Wallis test: The mean for each metal was determined and also used to calculate the Kruskal Wallis test. The Table 4 shows the constants and parameters from the calculation. From the table it would be seen that count of number of samples, total number of samples and degrees of freedom were the same, while the values of sum of rank differs. Accordingly, both result from the AAS and XRF were accepted because H statistics were lower than H critical. Thus there was a significant difference between the concentrations of metals analysed by of XRF and AAS spectroscopic analysis [15].

Hence we principally suggest that the AAS and XRF instrumentation showed different sensitivity and selectivity of metal samples from the recharge card of both networks [15]. Alternatively, we suggest that different suppliers produce their recharge cards or same supplier producing the recharge cards using different coating preparations. This result formed a correlation with discussion obtained from HRI, RfD, CV and THQ that showed variation even within same sampling points as shown in Fig. 2. The result also agreed with all metals in Fig. 1 (Al, Cr, Cu, Fe, Zn) with exception of Ag.

The Spearman rank correlation coefficient: The mean for each metal was also determined and similarly used for the calculation of the Spearman rank correlation coefficient. The Table 5 shows

the constants and parameters obtained from the calculation. From the table it would be observed that only critical value and number of data pairs were equal while the difference in ranks differs. Accordingly, both results from XRF and AAS were accepted because the  $r_s$  value was less than the critical value. Thus there was a significant difference in the concentration of metals analysed by both instrumentations.

Hence, we mainly suggest that both XRF and AAS instrumentation showed selectivity and sensitivity to metal concentrations from both networks [15]. Also alternatively it would be suggested that different producers were supplying the recharge cards using different concentrations or that same producer was producing the same recharge cards using different coating preparations. Additionally, it would be concluded that both the Spearman rank correlation coefficient and Kruskal Wallis test agreed with all the risk assessment index HRI, RfD, CV and THQ findings. The result also agreed with all metals in Fig. 1 (Al, Cr, Cu, Fe, Zn) with exception of Ag as previously discussed.

#### 4. CONCLUSION

The analysis showed that AAS had more sensitivity (than XRF) to metals analyzed except in Al and Ag. The concentration trend of metals

was Ag>Fe>Cr>Zn>Cu>Al with sampling point 1 detecting their highest concentrations. Both the THQ and HRI of all metals were below their reference value except Fe at unacceptable exposure levels. The correlation between spearman correlation and Kruskal Wallis test agreed with results obtained from HRI, Of, CV and THQ that showed variation even within same sampling points. On the Contrary, the Wilcoxon Mann Whitney test showed no variation, a pointer to Ag and Al unusual behavior as soluble metals in recharge cards waste.

### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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