Sensitivity and Lower Limit Detection Determined of XRF for Copper and Zinc Suspended in lubricated Grease

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Abstract

In this work different weight percent of copper and zinc powders of 20 µm particle size were blended carefully with grease , and the final weight of samples are 2 g . The samples were placed in containers were made from tephlon of 2.5 cm as a diameter . The bottom window of container was fitted with 6 µm mylar film . The samples were subjected to X- ray fluorescence to measure the Cu K_α and Zn K_α lines intensities . Sensitivity (m) and Lower Limit Detection (LLD) were calculated and plotted against the weight fraction for all samples .

Key words : XRF , Sensitivity ,Lower Limit Detection , Suspended particles

تحديد الحساسية والحد الأدنى للتحسس لتألق الأشعة السينية لنماذج من مساحيق النحاس والخارصين عالقة في شحوم التزييت .

الخلاصة

في هذا العمل تم خلط بعناية نسب وزنية مختلفة من مسحوق النحاس والخارصين ذات حجوم حبيبية 20µm مع شحوم المكائن بحيث يصبح وزن العينة 2g . وضعت النماذج في حاويات خاصة صنعت من التفلون بقطر 2.5cm . تم تغطية اسفل الحاوية المواجة للأشعة السينية بغشاء رقيق بسمك 6 µm وتم وضع النماذج في جهاز تألق الأشعة السينية لغرض قياس الشدة للخطوط Cu K_α و Zn K_α . تم حساب الحساسية والحد الأدنى للتحسس لتقنية تألق الأشعة السينية وتم رسم تغاير الحساسية والحد الأدنى للتحسس مع النسب الوزنية لكل من النحاس والخارصين .

Introduction

In recent years X-ray fluorescence (XRF) has been found to offer advantages of speed , accuracy and nondestructive means . The determination of Copper and Zinc particles in engine grease is established as a valuable means of assessing concentration of particles in grease [1,2] . XRF analysis represented as a method to assessing wear metal debris related to working process in different applications , this can be performed by making calibration curve of standard samples and compared with samples , and can be considered as a test method to evaluates the fretting wear protection provided by lubrication greases [3] . The large field of oil analysis of suspended particles , further more evaluates the lubricant for its condition and for the presence of other contaminates such as fuel , coolant , and water [4 ,5, 6] .

Theoretical part

Different parameters affected on XRF technique sensitivity and lower limit detection such as instrument component , accessories , X-ray background , and sample atomic number . The last parameter may be made chosen for optimum sensitivity and

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lower limit detection for a given X-ray tube target , diffracted analyzing crystal , collimator system , and detector type [7] . The sensitivity (m) of XRF spectrometer is determine by the following equation [8] .

$$m = \frac{count(c/s)}{concentration(\%)} \quad \text{-------(1)}$$

The generally accepted definition of lower limit detection(LLD) is that concentration equivalent to two standard deviations of background counting rate[8] . There are two standard deviations of the total background counts (N_b) taken will be given by :

$$2S = 2\sqrt{N_b} = 2\sqrt{R_B t_b} \quad \text{---- (2)}$$

Where S: standard deviation

t_b: the time spent counting on the background

R_b: the count of background

To convert count to count rate we dividing by time as follows :

$$2S = 2\frac{\sqrt{R_b t_b}}{t_b} = 2\sqrt{\frac{R_B}{t_b}} \quad \text{-------(3)}$$

To convert count rate to concentration we dividing by sensitivity (m) ahich is defined as :

$$2S = \frac{2}{m}\sqrt{\frac{R_b}{t_b}} \quad \text{----------(4)}$$

Since two measurements have to be made (peak and background) , the error increased be $\sqrt{2}$ and taking $2\sqrt{2}$. We have the formula of LLD

$$LLD = \frac{2}{m}\sqrt{\frac{R_b}{t_b}} \quad \text{----------(5)}$$

This equation represent the lower limit detection for the XRF technique type wavelength dispersive[9] .

Experimental part

A siemens SRS 200 sequential X-ray spectrometer complete with Kirstalloflex 805 X-ray generator and measuring cabinet with electronic measuring system and Kompensograph X-T . A molybdenum (Mo) tube target was used at the operating power of X-ray system was 30KV and 17mA . LiF (100) analyzing crystal , collimator of $0.^{o}15$, detector type is scintillation counter ,Vacuum of 10^{-3} m bar were used in both sample and analyzing chamber [5].

The samples were prepared by blending carefully different weight percentages form Copper and Zinc fine powders with a Calcium grease of code number C-2 and worked penetration at 25 ^{o}C is 265-295(without units) and the average drop point 95 ^{o}C according to [10] . The samples were placed in Tephlon containers and subjected to X-ray . Counts were accumulated for 10 second and averaged to 1 second to measured the XRF intensities of Cu Kα and Zn Kα lines at peaks and also for either side to obtain background . The average of background intensities were calculated to give net counts of Cu Kα and Zn Kα lines .

Results and discussion

The results of Cu Kα and Zn Kα lines intensity measurements as a peaks and background were tabulated in tables (1) and (2) , and by employing these results and equations (1) and (5) can be calculated the sensitivity (m) and lower limit detection (LLD) . The results of

sensitivity (m) were plotted versus with weight fraction of Cu and Zn as shown in figure (1) , and The results of lower limit detection (LLD)were plotted versus with weight fraction of Cu and Zn as shown in figure (2) . From figure (1) one can noticed that the optimum sensitivity at 0.5wt% for Cu and Zn which was represented the highest value of sensitivity . , and from figure (2) can be shown that best value (lowers value) of LLD at 0.5wt% for all samples . The values of sensitivity and lower limit detection indicates that can be use XRF technique at the approximate limit of 0.0028% and this value represent the minimum percent of metal particles suspended in grease which were detected by using X-ray fluorescence of conditions mentioned in experimental part .

Conclusion

1- The XRF intensity of Cu Kα and Zn Kα increased when the suspended particles concentration increased .
2- The Lower Limit Detection (LLD) and sensitivity (m) of XRF spectrometer depend on atomic number of suspended metallic particles in Grease .
3- The maximum sensitivity value of XRF at 0.5wt% for Cu and Zn , and the minimum LLD of XRF spectrometer technique at 0.5wt% for Cu and Zn suspended particles in Grease .

References:

[1]-Humphrey G.R., " Characterization of debris from F404 engine oil filters By energy dispersive X-ray Fluo ;JOAP- TSC-TR-96-02 . (1996) .

[2]-Whitlock R.R., "The path to affordable long term failure warning : The XRF- wear monitor" , (1998) .

[3]-Whitlock R.R., Restoring wear condition monitoring with XRF" predictive maintenance technology national conference -1996 , Scpublishing Co. , Minden , (1996) .

[4]- ASTM D417-87 , "Standard test method for fretting wear protection by lubricating Grease " ,(1987) .

[5]- Judran A.K., "Studies of X-ray fluorescence of suspended particles in Iraqi oil" Ph.D. Dept. of Physics , College of science , University of Baghdad , (2006) .

[6]-Bertin E. , "Principles and practice of X-ray spectrometric analysis" p92-97 , (1975) .

[7]-Jenkins R., " X-ray technique : encyclopedia of analytical chemistry p13269-13288 , publishers John wily and sons Ltd , (2000) .

[7]-Clark D. and Mark B. (2000) "Determination trace element in water using micro sample X-ray analysis" p92-97 .

[8]-White G.W., "ASTM data series DS-37A X-ray absorption wavelength and two theta tables" Second edition , (1970) .

[9]- Iraqi specification " Marketing specification of Iraqi petroleum production product , (2000) .

Table(1) The results of Cu Kα line intensity measurements as a peaks and background

Cu (wt%)	Cu Kα peak (c/s)	Cu Kα background (c/s)	Net count (c/s)
0.1	1200	150	1050
0.5	5770	170	5600
1	11600	200	11400
1.5	16140	240	15900
2	20760	260	20500

Table(2) The results of Zn Kα line intensity measurements as a peaks and background

Zn(wt%)	Zn Kα peak (c/s)	Zn Kα background (c/s)	Net count (c/s)
0.1	1450	160	1290
0.5	7190	190	7000
1	13250	250	13000
1.5	18300	300	18000
2	23840	340	23500

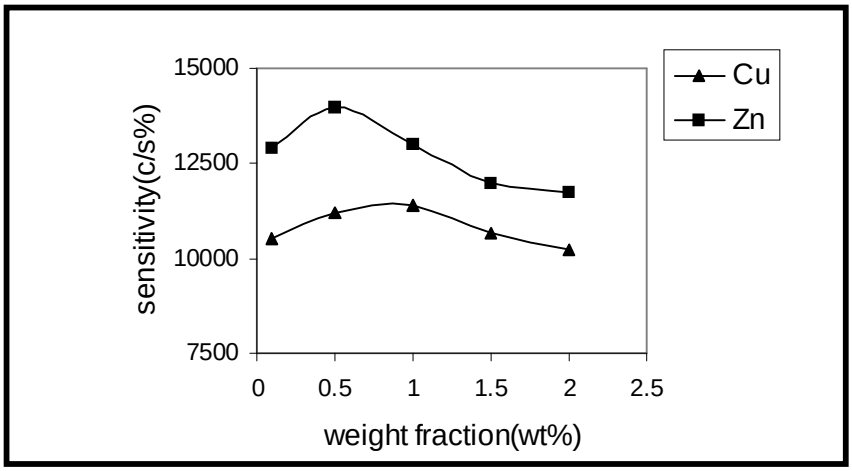

Fig.(1) Sensitivity as a function of weight fraction .

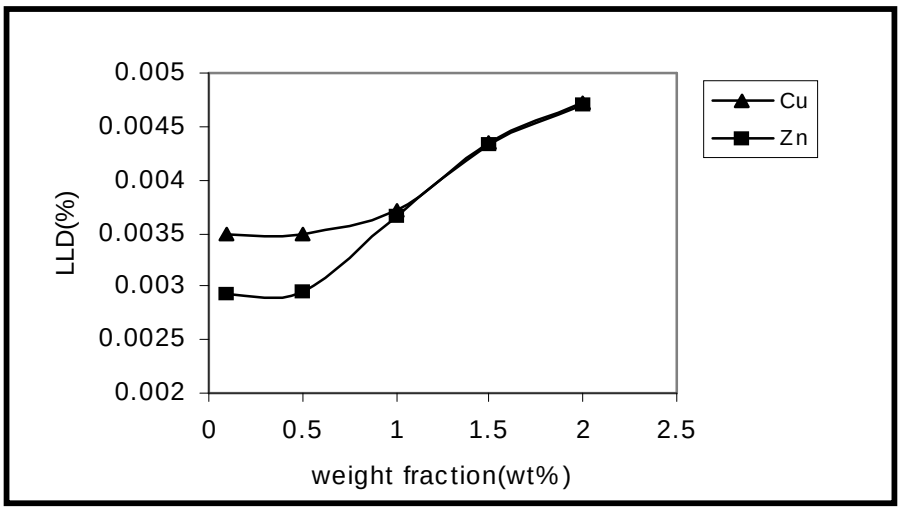

Fig.(2) Lower limit detection as a function of weight fraction .