Particle size effect on XRF measurements of Copper and Zinc particles suspended in hydrocarbon materials

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Abstract:

Copper and Zinc powders with different particle sizes were subjected to sieving of range (20-100μm) and He-Ne laser system to determine the particle size . 1wt% from each powders was blended carefully with 99wt% from Iraqi oil . Microscopic examination were carried for all samples to reveal the particle size distribution . XRF intensity measurements were conducted for all suspended samples , and the relation between XRF intensity and the particle size was found .

Introduction:

The utility of X-ray fluorescence (XRF) for analyzing metallic content in organic fluids has been recognized for along time , and has become the basis of the finger printing of oil sources [1,2] . The influence of particle size in the X- ray analysis of powders and homogenous solid has been evaluated methods of sample treatment which eliminate or minimize particle effect and in homogeneity have been proposed for suspended particles in oil , hence X-ray measurement of these suspensions was carried out to determine limitations of the X-ray fluorescence technique in such analysis [3,4] .

Theoretical part :

When X-ray pass through the object being tested , the signal is attenuated by scattering and absorption according to Beers law :

$I = I_O e^{-\mu\chi}$ ----------(1)

Where Io: the initial intensity .

I: the final intensity .

μ : the attenuation coefficient (cm-1) .

χ : the length of X-ray path (cm) .

But the actual volume of sample which can contribute to the measured fluorescent radiation is independent upon the effective penetration depth of the measured wave length [5,6] . This in turn supports need for completely homogenous specimen . Since if for instance compositional variation in depth are present if we consider a powder sample containing the element of interest in spherical particles of a uniform diameter D , then at any depth of χ from the surface there are likely to be N particles , where N is proportional to the concentration of the element . If the original particle diameter is reduced to (D/2) , Then at the same depth χ there will be (8N) particles , if the particles are cubic in shape then the particle size reduction again results in an eight fold increase in number and can be described as a lateral displacement of the lower half of the cubic as shown in Fig.(2.1a) . The exciting radiation is again absorbed by the matrix until it reaches the surface and is measured . Assuming the average depth of particles is the same for the different particle size , then the total matrix absorption is virtually

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independent of particle size . It is evident from Fig.(2.1b) that for cubic particles reducing the dimension of a side by two is equivalent to exposing twice the original area of the material being measured at a half of the original thickness . In the spherical particle the same is true since the surface area of a sphere is
proportional to the diameter squared . Thus , each of the large particle [7,8] . It is a well known fact that the intensity of a characteristic line emitted by a thin layer of material increases the thickness is increased up to a point which we will define as infinite thickness , value any increase in thickness will result in no observable gain in characteristic signal from the sample [9] . The relation ship between the fluorescent intensity from the same sample which of infinite thickness can be shown to be as follows [7,10] .

$(Ix/I\infty) = 1\text{-}EXP(-a\rho\chi)$ ----------(2)

Where

Ix and I∞ are the intensities of fluorescent radiation per square centimeter leaving samples of thickness χ and ∞ respectively .

a : the absorption parameter .

ρ : the density of sample .

χ : the thickness of sample .

and $a=\mu i \csc\theta i + \mu e \csc\theta e$ ----------(3)

Where

μi and μe are the absorption coefficient for incident and emergent X-ray respectively .

θi and θe are the incident and emergent analysis respectively .

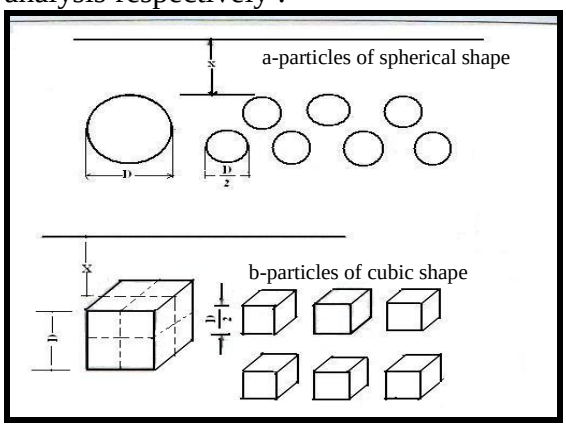

Fig.(2.1) Effect of particle size reduction .

Experimental part :

1 Particle size estimation :

The segregation of Iron , Cobalt ,and Nickel particles into size ranges by sieve and subjected to He-Ne laser system of wave length 632 nm and power of 1mwatt to determine the experimental average particle size for Cu and Zn powders with more accurate .

2 Sample preparation :

Table (3.1) represents sample code and their description for different particle size of Cu and Zn powders blended with Iraqi oil type Babel of SAE=40HD ,flash point = 236 , viscosity at 100 oC= 15 centistock , and pour point at -9 oC . 1wt% from each particles and 99wt% from oil were blended carefully manually and by using ultrasonic generator . Microscopic examination was conducted for all samples mentioned in table (3.1) to reveal the particle size distribution in oil matrix .

Table (3.1) Sample codes and their descriptions .

Sample code	Description
Cu_1O	0.1wt% copper with average particle size of 40μm+0.99wt% oil
Cu_2O	0.1wt% copper with average particle size of 55μm+0.99wt% oil
Cu_3O	0.1wt% copper with average particle size of 65μm+0.99wt% oil
Cu_4O	0.1wt% copper with average particle size of 80μm+0.99wt% oil
Zn_1O	0.1wt% Zinc with average particle size of 20μm+0.99wt% oil
Zn_2O	0.1wt% Zinc with average particle size of 30μm+0.99wt% oil
Zn_3O	0.1wt% Zinc with average particle size of 40μm+0.99wt% oil
Zn_4O	0.1wt% Zinc with average particle size of 60μm+0.99wt% oil

3 XRF measurements :

The XRF system was operated at fixed operation conditions of 30KV and 17mA . All samples which mentioned in table (3.1) were placed in samples container and subjected to X-ray fluorescence measurements . XRF intensities were in (count/10s) and averaged to (count/s) and conducted for all samples to evaluate the effect of particle size on X-ray fluorescence intensity.

Results and discussion :

Figures (4.1) and (4.2) show the microscopic examination of Copper and Zinc particles suspended in oil . These

figures reveal the differences in particle sizes distribution in oil matrix .

Figures (4.3) and (4.4) represent the XRF relative intensity (which was calculated as the peak intensity to the background intensity for the same sample) for Cu and Zn powders suspended in oil as a function of particle sizes . These figures exhibited the results of XRF measurements for different particle size . Particles of smallest size shown to yield highest intensities while particles which have large particle sizes shown to yield lowest intensities for all specimens , the behavior of intensity curve due to that the increase in intensity was attributed to a decrease in voids of the specimen surface with the reduction in particle size , and this leads to the excitation area will be large . This situation agrees with the relationship (2) between the XRF intensity and particle size.

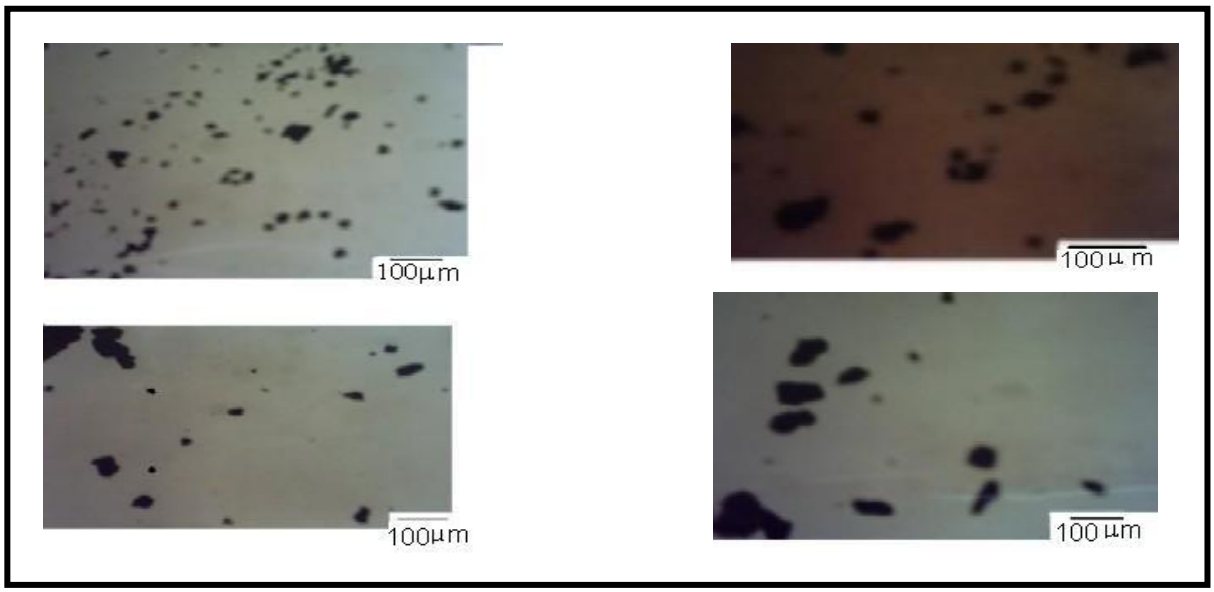

Fig (4.1) Microscopic photograph of Copper powder suspended in oil at different particle size .X100

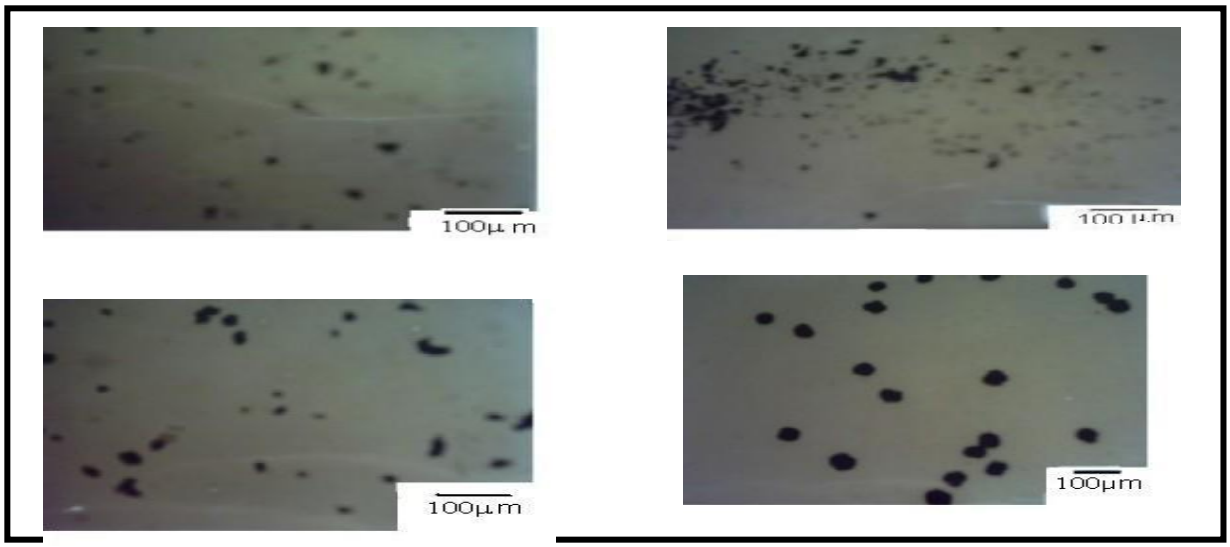

Fig (4.2)Microscopic photograph of Zinc powder suspended in oil at different particle size .X100

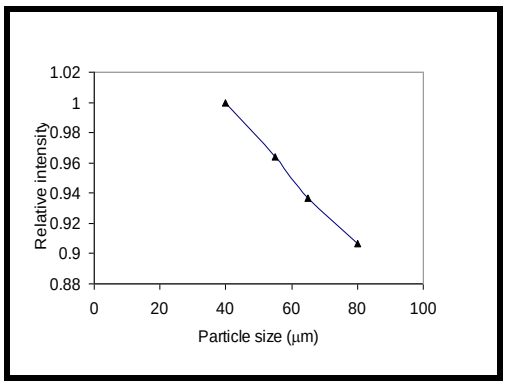

Fig.(4.3) XRF relative intensity of Cu as a function of particle size .

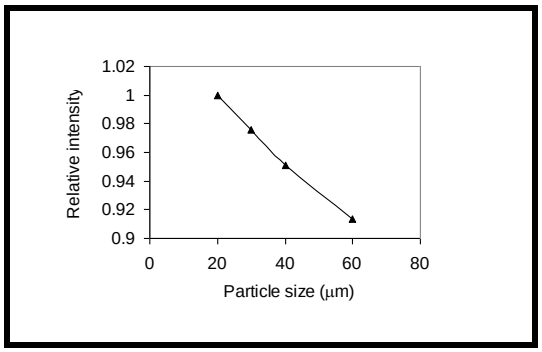

Fig.(4.4) XRF relative intensity of Zn as a function of particle size .

Conclusions :

1- The X-ray fluorescence intensities affected by particle size of different metal powders .

2- By using XRF technique can be evaluated the composition of suspended metal particles in liquid hydrocarbon .

3- The XRF intensities is high when the particle size is small , and vise versa . The relationship between them approach to take exponential form .

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و الزنك عالقة في المواد الهيدروكاربونية

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الخلاصة:
مساحيق من النحاس والزنك بمختلف الحجم الحبيبي أجريت عليها قياسات بواسطة الغرابيل بمدى (20-100μm) وليزر الهليوم – نيون لإيجاد الحجم الحبيبي . (1wt%) من هذه المساحيق خلطت بعناية مع (99wt%) من الزيت العراقي . تم إجراء الفحص المجهري لكل العينات لمعرفة توزيع الجسيمات في الزيت . أجريت قياسات شدة تألق الأشعة السينية لكل العينات ومن تم إيجاد علاقة بين شدة تألق الأشعة السينية وحجم الجسيمات .