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Differentiation of Fertilized and Non Fertilized Clay Sand Samples Using XRF and AAS Techniques

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Abstract

Plants are very important for human life. recently it was observed that plants using chemical fertilizers cause some times severe biological hazards. On the other hand, the elemental contents of the soil determine the suitable plants which can grow in it. This encourages to do this work which aims to determine the elemental contents of clay and sand soil besides studying the effect of adding chemical fertilizer to them.

In this work sixteen samples were prepared 5cm depth from clay soil region, four samples were non-fertilized and four samples were fertilized one were collected in a powder form. From sand soil region four samples were non-fertilized and chemical fertilized samples were also prepared.

The elemental contents of all samples were determined using x-ray fluorescence (XRF) and atomic absorption spectrometer (AAS) The results obtained showed that for clay samples the concentration of (Fe) is higher in non-fertilized samples while the concentration of (Cr,Mn, Ni, Au) were higher in fertilized samples. for sand samples the concentration of (Au,Pb) were higher in non-fertilized samples while the concentration of (Cr,Mn,Ni) were higher in fertilized samples .This means that fertilization process added more (Cr, Mn, Ni) to the soil. the XRF technique looks more accurate than AAS as for as it detects more elements with higher concentration (higher detection limit) than the other two techniques.

It can detect (Mo, W,V) in clay samples beside (Co,Ti,Nb) in sand samples which cannot be detected by AAS.
Thus in soil elemental analysis XRF is more suitable while AAS can confirm the results of XRF.

Introduction

X-ray fluorescence spectrometry is a powerful technique for elemental analysis. When it was introduced on a commercial scale about 30 years ago, the main research efforts were devoted to the developments of instrumentation and practical applications, with only moderate attention to fundamental conditions. Generally, the continuing interest in trace elements has stimulated the developments of several powerful analytical techniques for their detection and quantitative determination, one of these is X-Ray Fluorescence (XRF), where a source of x-ray photons is used to study the elemental composition of materials. Absorption of these photons by the

photoelectric effect produces vacancies in the inner electron shells of the atoms of material, followed by the emission of characteristic x-rays of the elements present.
AAS finds wide application in fields that vary from mining to pharmaceuticals, environmental control and agriculture. Most heavy metals are toxic and should be avoided as far as possible. If you ever had to use an antibiotic, chances are that the quality control process to ensure that the drug is free from the catalysts like palladium or platinum used to make them was performed by an AAS. Similarly, the food and health supplement industries make use of AAS to ensure that their products are safe for consumption. In mining, a lot of focus is on the recovery of precious metals like gold from old mining heaps. With the help of AAS, the amount of gold can be quantified to determine whether it would be profitable to extract it. The analysis of drinking water is probably one of the most important applications of AAS, especially in places where the environment is not properly cared for
The elemental contents of all samples were determined using x-ray fluorescence (XRF) and atomic absorption spectrometer (AAS) The results obtained shows that for clay samples the concentration of Fe is higher in non-fertilized samples while the concentration of Cr,Mn, Ni, Au is higher in fertilized samples. for sand samples the concentration of Aube is higher in non-fertilized samples while the concentration of Cr, Manini is higher in fertilized samples.

This means that fertilization process added more Cr, Manini to the soil. the XRF technique looks more accurate than AAS and LIBS as for as it detects more elements with higher concentration (higher detection limit) than the other two techniques.

Material and Method

Two deferent methods are used in analyzing the above samples so as to achieve maximum accuracy of detection.

Sample preparation test steps for XRF analysis

* 16 sample of clay and sand soil dried at room temperature for three days.
*1gram of each soil sample powder was weighed accurately for each sample.
* sample putted in the exposure cup and exposed by x-met 5000 10 seconds.

Results XRF AND AAS comparison

Table (1) comparison of concentration of XRF and AAS

Sample	Method	Fe	Cr	Mn	Ni	Au	Zn	Mo	Cu	W	V	Pb
1-1	**XRF**	69.87	1.04	3.04	0.01	3.4	3.88	_	_	_	_	7.34
	AAS	62.87	_	_	0.01	3.8	_	_	_	_	_	6.52
1-2	**XRF**	84.24	0.23	0.56	0.90	_	_	2.91	4.23	9.13	0.57	_
	AAS	81.27	_	_	0.90	_	_	_	4.14	9.14	_	_
1-	**XRF**	80.43	2.23	1.69	1.14	1.25	_	1.85	_	4.96	1.50	_

3												
	AAS	81.20	_	_	1.24	1.14	_	_	_	4.9	_	_
1-4	**XRF**	80.02	2.03	1.63	1.20	6.23	_	1.00	_	4.49	1.48	_
	AAS	79.20	-	_	1.22	6.25	_	_	_	4.4	_	_
2-1	**XRF**	75.66	0.55	1.60	2.50	2.32	_	1.11	_	7.55	6.24	_
	AAS	75.33	1.46	_	2.35	2.20	_	_	_	6.52	_	_
2-2	**XRF**	46.72	9.33	3.02	2.88	4.77	0.54	_	_	_	_	8.82
	AAS	42.33	9.36	_	2.80	4.73	_	_	_	_	_	8.76
2-3	**XRF**	73.95	0.55	9.42	0.90	2.23	_	2.26	_	8.23	1.65	_
	AAS	72.03	0.54	_	0.93	2.26	_	_	_	8.21	_	8.72
2-4	**XRF**	71.03	5.26	1.00	3.00	7.38	_	2.54	_	8.98	0.40	_
	AAS	62.87	5.25	_	3.01	7.36	_	_	_	8.96	_	-

Table (2)

Sample	Method	Fe	Cr	Mn	Ni	Au	Zn	Mo	Cu	W	V	C0	Ti	Nb	Pb
3-1	**XRF**	68.77	2.09	2.91	3.75	1.05	-	2.11	_	6.22	1.89				-
	AAS	67.87	2.06	_	3.77	1.08	_	_	_	6.21	_				6.52
3-2	**XRF**	80.09	2.09	0.92	3.33	0.21	_	5.00	-	-	0.31				_
	AAS	80.07	2.03	_	3.30	0.24	_	_	-	4.44	_				_
3-3	**XRF**	36.48	5.31	2.56	4.62	2.02	_	1.62	_	5.37	7.24		0.21		_
	AAS	37.20	5.62	_	4.64	2.04	_	_	_	5.39	_	-	-	-	-_
3-4	**XRF**	0.45	0.07	0.00	-	2.33		-	-	-	-	-	-	-	
	AAS	0.63	0.04			2.35	_	_	_	4.4	_	-	-	-	_
4-1	**XRF**	1.63	0.04	0.00	1.27	4.2	0.23	-	_	-	-	-	-	-	2.99
	AAS	22.03	0.06	_	1.33	4.01	_	_	_	-	_	-	-	-	2.82
4-2	**XRF**	80.37	0.00	0.90	0.59	5.43	-	1.70	_	8.62	0.93	-	-	-	3.01

	AAS	42.33	9.36	_	2.80	4.73	_	_	_	_	_				8.76
4-3	**XRF**	86.21	0.00	0.52	0.00	3.22	_	2.02	_	10.83	1.22	-	-	-	2.01
	AAS	85.33	0.00	_	0.35	3.20	_	_	_	10.72	_				
4-4	**XRF**	2.19	0.08	0.06	0.01	0.23	0.01	-	_						0.08
	AAS	80.7	0.00	_	5.551	5.36	_	_	_	8.55	_				-

Conclusion

Using three spectrometer techniques which are XRF, AAS and LIBS. The following was noticed. In view of tables (1) and (2) the average concentration of (Cr,Mn, Ni,Au) is more in fertilized than non-fertilized according to XRF and AAS results . However, the average concentration of Fe in non-fertilized clay samples is higher than fertilized. This may be attributed to the fact that clay, sand is rich in Fe while the fertilizers itself consists mainly of organic material thus is poor in (Fe). Thus it addition to clay samples to become fertilized decrease (Fe) concentration.
In some clay samples (Pb) appear but with higher concentration in fertilized clay samples according to XRF and AAS spectrometer results. This may be attributed to the fact that fertilizers are produced in side industrial areas which are highly contaminated with (Pb).

The results of LIBS for non-fertilized and fertilized clay samples gives concentration of almost all elements in the range of 8-12. there are no elements common for most fertilized and non-fertilized clay samples. Most elements detected by LIBS are absent in XRF and AAS results also most detected elements by XRF and AAS does not exist in LIBS. This make LIBS analyses less reliable and less accurate results. Table (1) shows the results of XRF and AAS for fertilized and non-fertilized sand samples. The average concentration of (Cr,Mn ,Ni) in fertilized sand are higher that of non-fertilized sand .However for (Au,Pb) the average concentration of them is higher in non fertilized sand compared to fertilized sand samples . This means that the fertilizer is poor in (Au) and (Pb) .The LIBS again gives less reliable and less accurate results as shown by table (2).Also comparing between XRF and AAS spectrometers indicates that XRF techniques gives more accurate results compared to AAS results. The XRF spectrometer detect elements like (Mo, W, V, Co, Ti, Nb) which cannot be detected by AAS .It is very interesting to note that some elements like (Co, Ti, Nb) are detected in sand samples only .The results of XRF, AAS and LIBS shows that the fertilization process only produce small change in the elements content of clay and sand samples. Sand samples consists of elements like (Co, Ti, and Nb) which does not exist in clay samples. The study indicates that XRF is more sensitive than AAS and LIBS techniques. The poorest technique is LIBS one.

*XRF is the most powerful technique for determination of concentration of elements.
*AAS can be utilized as a confirming technique to the XRF.
*The fact the use of chemical fertilizer does not change the chemical structure of the soil nessicates using organic fertilizers.

*Clay soil is rich in important vital elements thus are more suitable to be used for better plant growing.

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