Sudan University of Science and Technology
College of Graduate Studies

Detection of Some Elements in Sand (Reddish Orange and Black) by Using X-Ray Fluorescence Device

الكشف عن بعض العناصر في الرمل (البرتقالي المحمر والأسود) بإستخدام جهاز الأشعة السينية المتوهجة

Thesis submitted in partial fulfillment for requirement of the degree of master in physics

By

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قَالَ تَعَالَىٰ:

﴿ وَيَسۡـَٔلُونَكَ عَنِ ٱلرُّوحِۖ قُلِ ٱلرُّوحُ مِنۡ أَمۡرِ رَبِّي وَمَآ
أُوتِيتُم مِّنَ ٱلۡعِلۡمِ إِلَّا قَلِيلٗا ٨٥ ﴾

صدق الله العظيم

سورة الإسراء

Dedication

To the precious spirit … my mother

To my continues supporter … my father

To everyone who stood beside me and extended a helping, to my brothers, sisters and friends

Acknowledgement

My great Thank and my love to Allah who helps me to prepare this research.

I would like to thank the supervisor, Dr. Rawia Abdelgani Alobaid.

I offer all Thanks, appreciation and respect to Mr. Mohammed Abdelaziz Mohammed Elhassan for his benevolence and patience.

Abstract

This research deals with one of the applications of spectroscopy, which is the detection of some components of sand and the concentrations of these elements using X-ray fluorescence technology and comparison between them. Where sand samples were taken from Bara north Kordofan region (red-orange, black) from surface and depth (30cm, 70cm).

It was found that the elements present on the surface of the red-orange sample are:

Silicon (Si), Zirconium (Zr), Thorium (Th), Titanium (Ti), and their concentrations respectively (18.5%- 3.1%- 4.8%- 6.2%). And at depth 30cm found the same element (Si- Zr- Th- Ti) but with a different focus and it is (8.12%- 0.2%- 5.22%- 12.4%).

And black sand sample at the surface were the elements that contained it (Si- Zr- Th- Ti) and their concentrations respectively (32.3%- 3.02%- 9.25%- 14.01%). At depth 70cm an iron element (Fe) was found to add to these elements (Si- Zr- Th- Ti-Fe) concentrated also respectively (2.21%- 2.26%- 7.20%- 8.45%- 7.66%).

المستخلص

يتناول هذا البحث إحدى تطبيقات علم الأطياف و هي الكشف عن بعض العناصر المكونة للرمل وتراكيز هذه العناصر بتقنية الاشعة السينية المتوهجة والمقارنة بينهم .حيث تم أخذ عينات الرمل من منطقة بارا شمال كردفان (الرمل الأحمر-البرتقالي والأسود) من السطح وبعمق (30 سم و 70 سم).

ووجد أن العناصر الموجودة في السطح لعينة الرمل الأحمر-البرتقالي هي: السيلكونٍ (Si)، الزركنيوم(Zr)، الثوريوم (Th) ، التيتانيوم (Ti) وتراكيزها على التوالي (18.5% - 3.1% - 4.8% -6.2%) . وعند عمق 30 سم وجدت نفس العناصر(Ti- Th - Zr - Si) ولكن بتراكيز مختلفة وهي (8.12% - 0.2% - 5.22% - 12.4%) .

وعينة الرمل الأسود عند السطح كانت العناصر التي تحتويها (Ti - Th - Zr- Si) وتراكيزها (32.3% - 3.02%- 9.25%- 14.01%) على التوالي. وعند العمق 70 سم وجد عنصر الحديد (Fe) إضافة لتلك العناصر (Fe– Ti– Th – Zr – Si) بتراكيز ايضا على التوالي (2.21% -2.26%- 7.20%- 8.45%- 7.66%).

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CHAPTER ONE

INTRODUCTION

1.1 Preface

Sand is one of the most important resources found in nature-after air and water-so it has more than a precise definition. In generally is loose granular material that results from the disintegration of rock, consists of particle smaller than gravel but coarser than silt.

The exact definition of sand varies. The Scientific Unified Soil Classification System used in engineering and geology correspond to US stand sieves and defines sand as particles with diameter of between 0.074 and 4.75 millimeter. The sand varies in color, including white, black, orange-red, gray-white and other. According them different in using some of it is uses agriculture, construction, cement, beach restoration, glass and other.

In this research have identified some of the constituent elements of two types of sand using X-ray fluorescence device. X-ray fluorescence device is device that analyzes elements in general, such as qualitative and quantitative analysis. The dispersion of energy for x-ray fluorescence dispersion is one of the simplest and most accurate analytical methods for determining the chemical composition of many types of substances. It will also examine the study and practical analysis of samples of sand (black and red-orange) from Bara (city in west Sudan) to know the constituent elements and its concentrations.

1.2 Literature Review

- Mohamed Alzubir, Mohamed Ezeldin, Said Milad, (November, 2017), their study was to silicon dioxide and microcline, samples were collected from the north kordofan state Bara area, River Nile state Elmtama Area and Karema. Then samples were subjected to X-Ray diffractometer “XRD” according to standard test Method. Elements composition was characterized for all samples by X-Ray fluorescence spectrometer “XRF”. The particles size was carried out using sieve standards samples were acidified to improve the quality of silica. Silicon dioxide of Bara sand was found to be 100% while that of Elmtama was 96.6% silicon dioxide and 3.4% microcline standand for karima sand 94% silicon dioxide and 6% microcline. The percentages of the silica sand were found to be 95.8% for Bara, 90.9% for Elmtama and 8402% for Karema. The iron oxide percentage for Bara was found 0.03%.the particle size for Bara, Karema and Elmtama were found to be (60,100,140mm) respectively) [1].
- Dhruval Patel, Vivek Deshpande, Ela Jha, Viral Patel , (June, 2017), their study was to quality of castings in green sand mound is influenced by its properties such as green compression strength, green shear strength, permeability. The relations of these properties with the input parameters like sand grains size, shape, binder, clay is complex in nature. Binders play a vital role on green sand mould to enhance specific mould properties. The mould properties such as compression strength, permeability, hardness & shear strength have been studied & comparison have made with different binders. As per the study, for steel casting, they require 3% to 5% moisture content and 7% to 9% clay content respectively. Cause and effect diagram has been used to identify the different caused for casting defects. For optimizing the mould properties, Taguchi parametric design is studied and L9 orthogonal array is selected to find out optimal solution [2].
- Madhav Prasad Koirala, Er. Buddhi Raj Josh, (July, 2017), their study was to sand quality and cost-effective leads in rural village, a fundamental questions are identification of sources of construction sand mines, available quality and

supply management how to maintain it is big challenges? Should the license holder suppliers or a government agency above them mange the construction of the infrastructure project? To answer this question, we surveyed all society of consulting Architectural and Engineering Firms, Nepal (SCAFE) members involved in infrastructure projects, As per Federation contractors Association of Nepal (FCAN), sand quarry operator, sand suppliers and sand washing plant operators in Kathmandu valley of Nepal. Findings clearly indicate that the most important causes were lack of proper management, a warrens and understanding, quality, test skill and knowledge with contractor, consult and other stake holders. Also government and authority have not taken action to support legal aspects and control illegal operation as well as maintain supply management of construction sand for infrastructure projects [3].

1.3 Research Problem

Most of sand contains many elements that may different from sample to another according to the environment and types of sand and its quality.

1.4 Research Significance

Due to importance of sand in human life, this research studying two types of sand (orange-red and black) in terms of composition of the element and their concentrations at the surface and a certain depth to benefit from them in industries after processing.

1.5 Objective of Research

- Identification of some elements and their concentrations in orange- red sand from surface and depth 30 cm.
- Identification of elements and their concentrations in black sand from

Surface and depth 70cm.

- Comparing between the results in four samples.

1.6 Thesis Layout

This thesis is consist of four chapters, chapter one introduction and literature review, chapter two consists the basic concepts of spectroscopy, and its applications, the sand, X-Ray Fluorescence , chapter three consist methodology (materials, device and method) and result, chapter four consist of discussion, conclusion and recommendations, finally a list of references.

CHAPTER TWO

BASIC CONCEPTS

2.1 Sand

Sand is mixture of small grains of rock and granular materials which is mainly defined by size, being finer than gravel and coarser than silt. And ranging in size from 0.06 mm to 2mm. particles which are larger than 0.0078125 mm but smaller than 0.0625 mm are termed.

Sand is made erosion or broken pebbles and weathering of rock, which is carried by seas or rivers. And freezing and thawing during the winter break rock up the sand will be made. Sometimes sand on beaches can also be made by small broken-up pieces of coral, bone, and shell, which are broken up by predators and then battered by the sea, and even tiny pieces of glass from bottles discarded in the sea and other mineral materials [3].

2.1.1 Colors of sand

There are some different colors is found in sand. They are

1- **White sand**: it is made of eroded limestone and may contain coral and shell fragments, in addition to other organic or organically derived fragmental material is may find in this colors of sand.

2- Black sand: Black sand is composed of volcanic minerals and lava fragments and coral deposits.

3- Pink sand: Foraminifera, a microscopic organism that has a reddish-pink shell, is responsible for all this color found into this mix.

4- Reddish orange Color: this color is formed due to the coating of iron oxide.

5- White-grey Color: this sand consists of fine rounded grains and it is well graded.

6- Light-brown Color: It consists of rounded grains.

2.1.2 Classification of Sand

It is true people do not wonder about the origin of sand. Thousands of years need to pass for rocky material to finally turn into sand or clay. The sand that from sandstone rock, deposited as a beach, dune or desert. After millions of years, sandstone rocks turned into sandstone cliffs and eventually eroded for the second time. I noticed the differences in the various kinds of sand, from beaches, river, dunes, mountains, deserts and also from sand pits or quarries. Generally sand can be classification into categories from different project [3]:

Sand’s origin point of view, composition point of view and Grain size point of view.

Under origin point of view sand may divide to four sub categories

i. River sand
ii. Pit sand
iii. Marine sand
iv. Sand dune

Sand dune Under composition point of view, sand may divide to three sub categories

i. Clean sand
ii. Silt sand
iii. clayey sand

Under Grain size point of view, we can divide into 3 sub categories

i. Course sand
ii. Medium sand
iii. Fine sand

2.1.3 Using Sand

Sand is massively used for concreting, sand is used for preparation of mortar to bind with brick or stone or other materials, sand is used for preparation of mortar to plaster and pointing on wall surface, sand is massively used for concreting [3].

Hydropower work

a- Catchment
b- Dam Construction
c- Power house and much more

Road work, side drain work, seal coat work, pre-mix asphalt concrete work, pavement work. Canal work, Damp proof work, Tar felt work. Special work of sand: Sand is used for glass production, Sand is used for abrasive in sand blasting, Filtering water, Brick manufacture plant, Sand bags are used for protection against the flood, Sand castle building is popular activity for competition, Sand animation is a type of performance art, Aquaria are lined by sand, instead of gravel since it will be low cost, Rail road's use sand to improve the traction of wheels on the rails. Sandy soil will be ideal soil for some crops like watermelon, peaches and peanuts [3].

2.2 Spectroscopy

Spectroscopy is the branch of science which deals with techniques used to observe molecules. By using the physical properties of molecules and their structure, we can obtain information that indicates precise fork of the molecules. It is known study of the interaction between matter and light (electromagnetic radiation).

Light is a type of electromagnetic radiation consisting of little packets of energy called photons with both particle and wave-like properties, As shown in the complete electromagnetic spectrum in Figure (2.2), light in the visible region (~ 400 to 700 nm) make up only a small region of the entire spectrum of electromagnetic radiation [4].

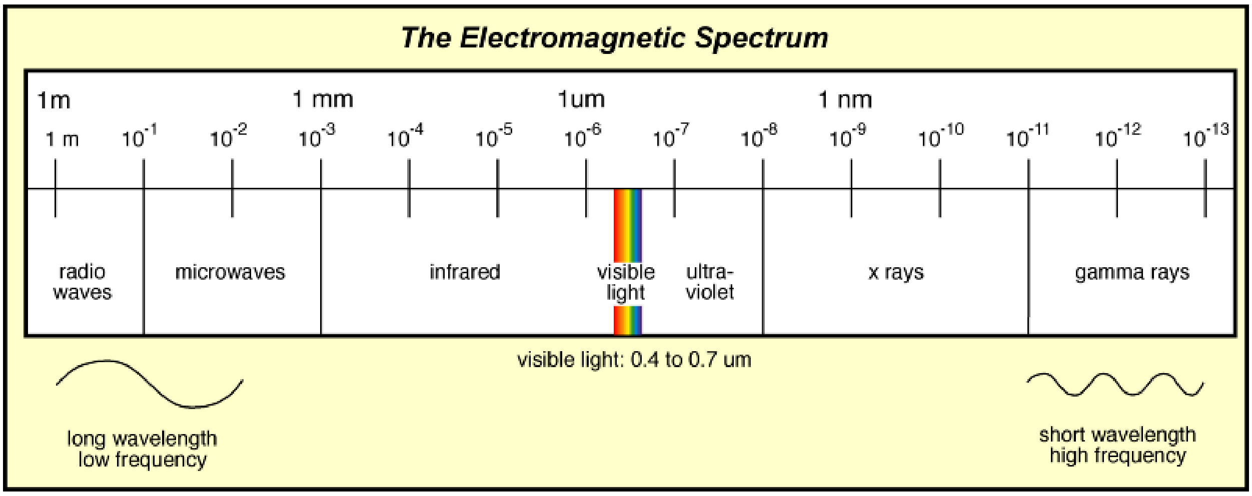

Figure 2.1: Electromagnetic Spectrum

It is the wave properties of light that limit ability to use light to create images. For any given wavelength, images can be formed for objects large than the wavelength of light used to visualize the image. Therefore, for visible light (wavelengths between $4{\times}10^{-7}$ and $7{\times}\ 10^{\ -7}$m) it is impossible to form images of atoms with sizes on the order of 10^{-9} m.

2.2.1 Wavelengths and energy

The energy of photon is inversely related to its wavelengths. The shortest wavelength (higher energy of the photon)

$$E = hc\ /\ \lambda \qquad (2.1)$$

Where:

E= Energy of the photon in joules

λ =wavelengths in nanometers.

h= plank constant ($6.62{\times}\ 10^{-24}$ J.s).

C=speed of light ($2.99782{\times}10^{-8}$m/s) [4].

2.2.2 The interaction of light with matter

It is the electrons in atoms and molecules that typically absorb and emit photons of light. All electrons have a series of energy levels they can occupy. The lowest energy level is referred to as the "ground state". The highest is the "ionization energy" or the energy required to completely remove the electron from the influence of the nucleus. In order for an electron to move from one level to a higher level it must absorb energy equal to the difference in the levels [4].

2.3 Types of spectroscopy

There are two distinct aspects of this interaction that can be used to learn atoms and molecules. One is the identification of the specific wavelengths of light that interact with the atoms and molecules. The other is the measurement of the amount of light absorbed or emitted at specific wavelengths. Both determination require separating a light source into its component wavelengths. Thus, a critical component of any spectroscopic measurement is breaking up of light into a spectrum showing the interaction of light with the sample at each wavelength.

Light interacts with matter in many ways. Two of the most common interactions are light that is absorbed by the atoms and molecules in the sample and light that is emitted after interacting with the atoms and molecules in the sample.

❖ Absorption Spectroscopy

Absorption spectroscopy is the study of light absorbed by molecules. For absorbance measurements, white light is passed through a sample and then through a device that breaks the light up into its component parts or a spectrum. White light is a mixture of all the wavelengths visible light. When white is passed through a sample, under the light conditions, the electron of the sample absorb some wavelength of light. This light is absorbed by the electrons so the light coming out of the sample will be missing those wavelengths corresponding to the energy levels of the electron in the sample.

❖ Emission Spectroscopy

Emission spectroscopy is the opposite of absorption spectroscopy. The electrons of the sample are promoted to very high energy levels by any one of a variety of method (e.g., electric discharge, heat, light, etc.). As these electrons return to lower levels they emit light [4].

2.4 Other types

Other types of spectroscopy are distinguished by specific applications or implementations:

- Acoustic resonance spectroscopy is based on sound waves primarily in the audible and ultrasonic regions
- Auger spectroscopy is a method used to study surfaces of materials on a micro-scale. It is often used in connection with electron microscopy.
- Cavity ring down spectroscopy
- Circular Dichroism spectroscopy
- Coherent anti-Stokes Raman spectroscopy (CARS) is a recent technique that has high sensitivity and powerful applications for *in vivo* spectroscopy and imaging.
- Cold vapor atomic fluorescence spectroscopy
- Correlation spectroscopy encompasses several types of two-dimensional NMR spectroscopy.
- Dual polarization interferometry measures the real and imaginary components of the complex refractive index
- Electron phenomenological spectroscopy measures physicochemical properties and characteristics of electronic structure of multicomponent and complex molecular systems.
- EPR spectroscopy
- Force spectroscopy
- Fourier transform spectroscopy is an efficient method for processing spectra data obtained using interferometers. Fourier transform infrared

spectroscopy (FTIR) is a common implementation of infrared spectroscopy. NMR also employs Fourier transforms.

- Hyperspectral imaging is a method to create a complete picture of the environment or various objects, each pixel containing a full visible, VNIR, NIR, or infrared spectrum.
- Inelastic electron tunneling spectroscopy (IETS) uses the changes in current due to inelastic electron-vibration interaction at specific energies that can also measure optically forbidden transitions [5].
- Inelastic neutron scattering is similar to Raman spectroscopy, but uses neutrons instead of photons.
- Laser-Induced Breakdown Spectroscopy(LIBS), also called Laser-induced plasma spectrometry (LIPS)
- Laser spectroscopy uses tunable lasers and other types of coherent emission sources, such as optical parametric oscillators, for selective excitation of atomic or molecular species.
- Mass spectroscopy is an historical term used to refer to mass spectrometry. Current recommendations are to use the latter term. Use of the term mass spectroscopy originated in the use of phosphor screens to detect ions.
- Neutron spin echo spectroscopy measures internal dynamics in proteins and other soft matter systems
- Photoacoustic spectroscopy measures the sound waves produced upon the absorption of radiation.
- Photoemission spectroscopy
- Photothermal spectroscopy measures heat evolved upon absorption of radiation.
- Pump-probe spectroscopy can use ultrafast laser pulses to measure reaction intermediates in the femtosecond timescale.

- Raman optical activity spectroscopy exploits Raman scattering and optical activity effects to reveal detailed information on chiral centers in molecules.
- Raman spectroscopy.
- Saturated spectroscopy
- Scanning tunneling spectroscopy
- Spectrophotometry
- Spin noise spectroscopy traces spontaneous fluctuations of electronic and nuclear spins [5].
- Time-resolved spectroscopy measures the decay rate of excited states using various spectroscopic methods.
- Time-Stretch Spectroscopy.
- Thermal infrared spectroscopy measures thermal radiation emitted from materials and surfaces and is used to determine the type of bonds present in a sample as well as their lattice environment. The techniques are widely used by organic chemists, mineralogists, and planetary scientists.
- Ultraviolet photoelectron spectroscopy(UPS)
- Ultraviolet–visible spectroscopy
- Vibrational circular dichroism spectroscopy
- Video spectroscopy
- X-ray photoelectron spectroscopy (XPS) [5].

2.5 Applications of spectroscopy

- Cure monitoring of composites using optical fibers.
- Estimate weathered wood exposure times using near infrared spectroscopy.
- Measurement of different compounds in food samples by absorption spectroscopy both in visible and infrared spectrum.

2.6 X-Ray fluorescence

X-ray fluorescence method that can determine concentration of major, minor and, in some cases, trace elements by using x-rays. The most common XRF set-up used in heritage laboratories is energy dispersive (ED) XRF. During an ED-XRF experiment, a high-energy X-ray beam hits the surface of the objective or sample under observation. This in turn emits x-ray of lower energy, behavior known as “fluorescence”.

2.6.1 Principle of X-Ray fluorescence

In XRF, x-ray produced by a source irradiate the sample. In most cases, the source is an X-ray tube but alternatively it could be a radioactive material. The elements present in the sample will emit fluorescent X-ray radiation with discrete energies (equivalent to colors in optical light) that are characteristic for these elements. A different energy is equivalent to a different colors. By measuring the energies (determining the colors) of the radiation emitted by the sample it is possible to determine which elements are present. By measuring the intensities of the emitted energies (colors) it is possible to determine how much of each element is present in the sample. This step is called quantitative [6].

Each chemical element produces fluorescence X-rays with energies that are unique to that element. The emitted X-ray are displayed as a spectrum with peak can usually be linked to the abundance of the element in the sample.

2.6.2 Interaction of X-rays with matter

When X-ray beam passes through matter, some photons will be absorbed inside the material or scattered away from the original path. The intensity I0 of an X-ray beam passing through a layer of thickness d and density ρ is reduced to an intensity I according to the well-known law of Lambert-Beer:

$$I = I_0 \, e^{-\mu\rho d} \qquad (2.2)$$

The number of photons (the intensity) is reduced but their energy is generally unchanged. The term μ is called the mass attenuation coefficient and has the dimension cm2/g. The product μL = μρ is called the linear absorption coefficient and is expressed in cm-1. μ (E) is sometimes also called the total cross-section for X-ray absorption at energy E.

The mass absorption coefficient μ plays a very important role in quantitative XRF analysis. Both the exciting primary radiation and the fluorescence radiation are attenuated in the sample. To relate the observed fluorescence intensity to the concentration, this attenuation must be taken into account. Cumulative effect of several types of photon-matter interaction processes that take place in parallel. Accordingly, in the X-ray range the mass attenuation coefficient μi of element

i can be expressed as [5]:

$$\mu_i = \tau_i + \sigma_i \tag{2.3}$$

Where τ_i is the cross-section for photo-electric ionization and σ_i the cross-section for scattering interactions. All above-mentioned cross-sections are energy (or wavelength) dependent. Except at absorption edges (see below), μ is more or less proportional to Z 4λ3.

This method can be used to detect and quantify elements within radioactive materials. The polycarpellary focusing optics act as spatial filters to eliminate background radiation from the sample and increase detection sensitivity for sample elements of interest. Additionally, confocal XRF can be used for elemental depth profiling. Confocal XRF acts as a material probe by exciting and detecting emitted characteristic X-ray photons from within the confocal analysis volume as this volume is through the sample. In this way elemental

Concentrations can be measured on the object's surface and throughout the

Object's interior. Confocal X-ray fluorescence with X-ray optics has been used for many unique applications including thin film thickness measurement, forensics, art restoration, and small feature analysis [5].

CHAPTER THREE

METHODOLOGY AND RESULTS

3.1 Introduction

The aim of this chapter was to show the materials, apparatus and method that used in this work (samples preparation and setup).

3.2 Materials

Four samples of sand (reddish orange sand from Surface, reddish orange sand from depth 30Cm**,** black sand from surface black sand from depth 70Cm), we re prepared to identify them by X-ray fluorescence.

Figure 3.1: Sample of reddish orange sand.

Figure 3.2: Sample of black sand.

3.3 Apparatus

XRF device used to analyze samples that by stimulate the atoms inside and determined the types of elements within the samples.

Figure 3.3: X-ray fluorescence (XRF) device.

3.4 Method

Orange-red sand from Surface, reddish orange sand from depth 30cm, black sand from surface, black sand from depth 70cm, were prepared and subjected to XRF device to identify some of the elements that make up the samples.

3.5 Results of Identification of samples by X-ray fluorescence:

Table 3.1: Result of reddish orange sand from surface.

Elements	Concentrations%	STD
Si	18.5	0.136
Zr	3.1	0.046
Th	4.8	0.011
Ti	6.2	0.006

Table 3.2: Result of reddish orange sand from depth 30Cm.

Elements	Concentrations%	STD
Si	8.12	0.136
Zr	0.2	0.046
Th	5.22	0.011
Ti	12.4	0.0

Table 3.3: Result of black sand from surface.

Elements	Concentrations%	STD
Si	32.3	0.136
Zr	3.02	0.046
Th	9.25	0.011
Ti	14.01	0.006

Table 3.4: Result of black sand from depth 70Cm.

Elements	Concentrations%	STD
Si	12.21	0.136
Zr	2.26	0.046
Th	7.20	0.011
Ti	8.45	0.006
Fe	7.66	0.004

CHAPTER FOUR

DISCUSSION AND CONCLUSION

4.1 Introduction

This Chapter obtains the discussion of results and conclusion and recommendations.

4.2 Chart of Result

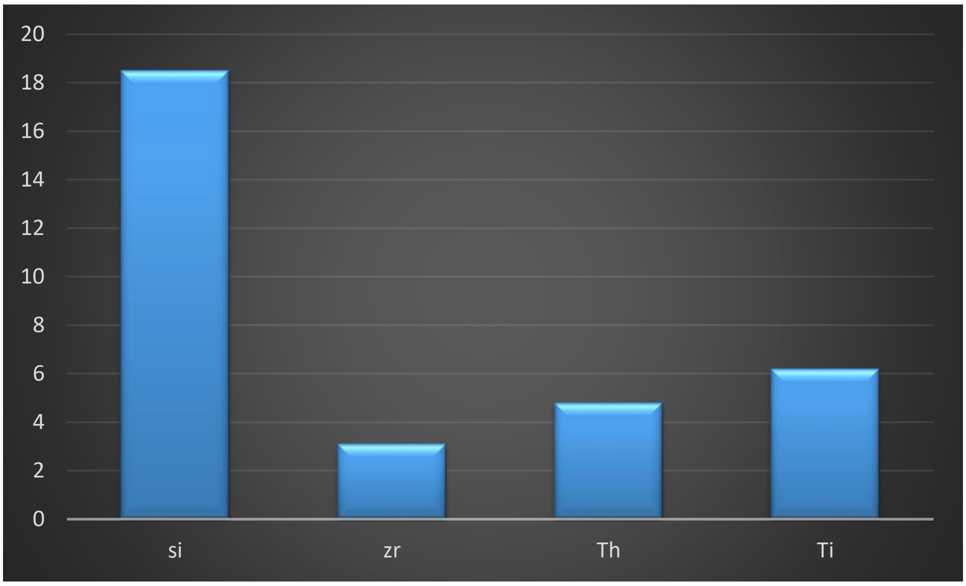

Figure 4.1: shows the analyze sample of reddish orange sand from surface by XRF.

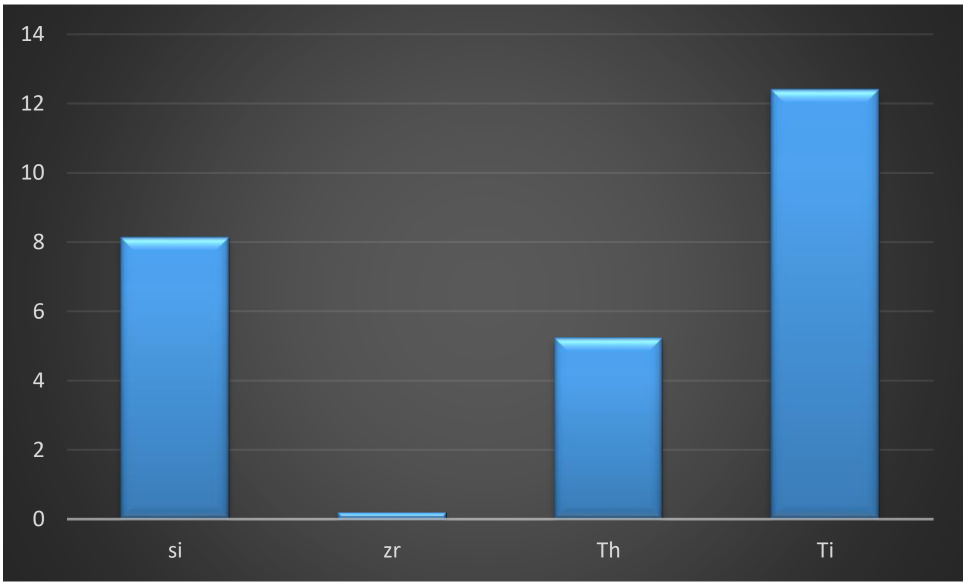

Figure 4.2: shows the analyze sample of reddish orange sand from depth 30Cm by XRF.

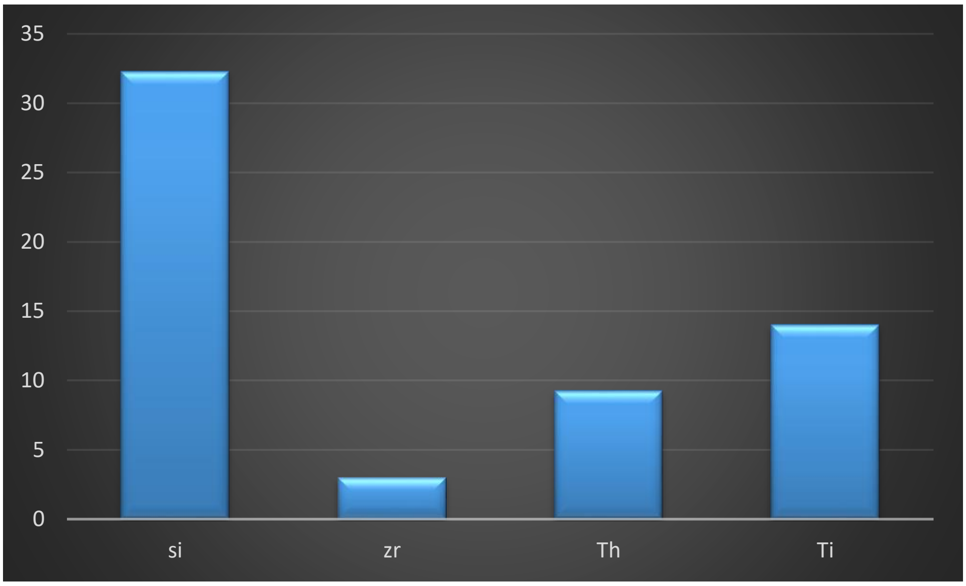

Figure 4.3: shows the analyze sample of black sand from surface by XRF.

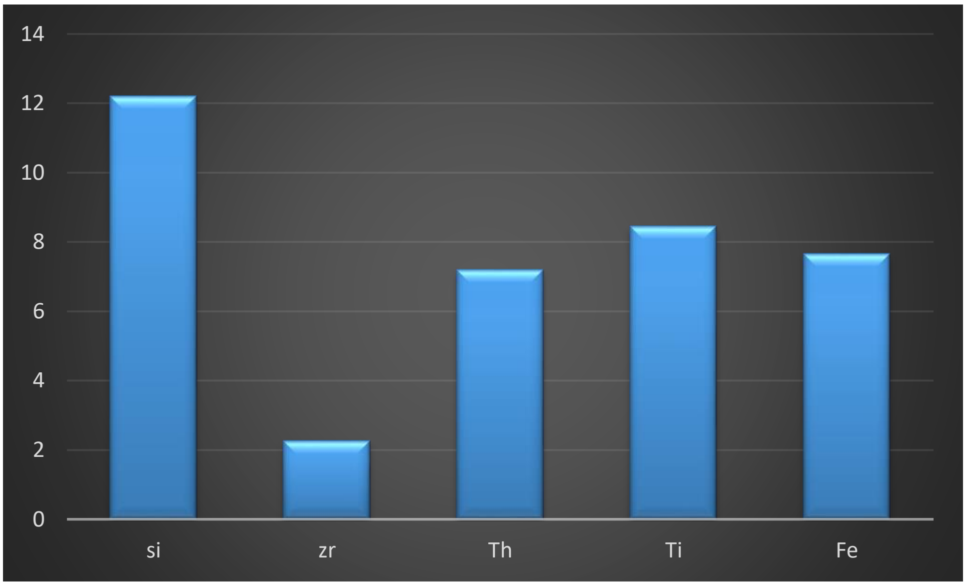

Figure 4.4: shows the analyze sample of black sand from depth 70Cm by XRF.

4.3 Discussion

The elements in four samples were found in different concentrations; which in reddish orange sand from surface found Silicon element, its concentration was 18.5%, Zirconium element and its concentration 3.1%, Thorium element and its concentration was 4.8% and Titanium element and its concentration 6.2%.

In reddish orange sand from depth 30Cm found Silicon element, its concentration was 8.12%, Zirconium element and its concentration 0.2%, Thorium element and its concentration was 5.22% and Titanium element and its concentration 12.4%.

In black sand from surface found Silicon element, its concentration was 32.12%, Zirconium element and its concentration 9.25%, Thorium element and its concentration was 5.22% and Titanium element and its concentration 14.01%.

In black sand from depth 70Cm found Silicon element, its concentration was 12.21%, Zirconium element and its concentration 2.26%, Thorium element and its concentration was 7.20%, Titanium element and its concentration 8.45% and Iron element and its concentration was 7.66%.

4.4 Conclusion

Sand contains important elements that distinguish it with very beneficial properties in human life, such as agriculture, cement, building clay, Industrial Island and glass, as a comparing between reddish orange sand and black sand were found the concentrations of elements in black sand were greater than reddish orange sand in two position (surface and depth). In black sand in depth 70cm were found the elements concentrations greater than black sand in surface, but in reddish orange sand were found that their elements concentrations in depth 30cm were greater than surface except two elements (Th, Ti) due to that they are heavy elements, and in depth 70cm in black sand the iron element was found with concentration 7.66%.

4.5 Recommendations

The researchers or furthers studies in this research problem could be done using another devices; such as X-Ray Diffraction (XRD), UV-Vis spectrometer, Raman spectroscopy, Fourier Transformer Infra-Red (FTIR), also could be done using XRF also but to identify another samples, and separate the elements that make up the sand to use for any applications that may use them.

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