Sudan University of Science and Technology

College of Graduates Studies

Determination of Trace Elements in Pure Water, Red and Green Tea and Their Drinking's Using Laser Induced coupled Plasma spectrometer

إيجاد العناصر في الماء الصافي والشاي الأحمر والأخضر ومشروباتهما باستخدام مطياف البلازما المقترن المستحث بالليزر

A thesis submitted for the partial Fulfillment of the Degree of Master in physics

By:

Hamza El sheikh Altyb Altahir

Supervisor:

Prof. *Mubarak Dirar Abd-Alla Yagoup*

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Abstract

Contamination of water drinks and food by toxic elements affect severely human health the aim of this work is to determine the sources of these contaminants in some tea drinking's. To do this 5 samples were prepared and examined. Three of them are pure water, row red and green tea, the other two are red tea water and green tea water drinking's. the elemental composition of the (5) samples was determined using laser induced coupled plasma (ICP) spectrometer. The tea drinking's were prepared by filter paper extraction. The results obtained shows that the contaminant nonorganic elements come from water, row tea, containers or surrounding environment.

المستخلص

تؤثر عملية تؤثر الماء والمشروبات والطعام لملوثات العناصر السامة على صحة الإنسان بشكل خطير. لذا تهدف هذه الدراسة لتحديد مصادر هذه الملوثات في بعض مشروبات الشاي. لعمل ذلك حضرت (٥) عينات وتم اختبارها. ثلاثة منها هي الماء وخام الشاي الأحمر والأخضر بالإضافة لعينتين هما مشروب الشاي الأحمر والأخضر. تم التعرف على مكونات العينات من العناصر باستخدام مطياف انبعاث الليزر المقترن بالبلازما. تم تحضير مشروبي الشاي الأحمر والأخضر باستخلاصه بمرشح ورقي. بينت النتائج أن العناصر الملوثة الغير عضوية تأتي من الماء وخام الشاي والأواني والبيئة المحيطة.

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Chapter One

Introduction

1.1 Spectral Techniques:

Dangerous chemical compounds recently pays attention of scientists for their severe effects on human health. This is since it can cause cancer or renal diseases as well as heart and hypertension diseases. This encourages researchers to use modern techniques so as to detect the existence of these dangerous chemicals. chemicals These techniques are based on the atomic structure which was predicted by the laws of quantum physics. Spectral techniques are recently used for mineral exploration in rocks. They are also widely used to determine the concentration of elements in soils as well as trace elements in plants and living organisms tissues.These techniques can be used to detect the dangerous chemical compounds.compounds.This is because such techniques are more accurate and has higher sensitivity. There are many spectral techniques like atomic absorption spectrometer(AAS), x-ray fluorescence and laser-induced plasma sensitivity. One of the most popular known new technique is the optical lastechni1 [1,2,3].

The optical emission spectroscopy of laser-induced plasma is named as laser induced break down spectroscopy (LIBS) or sometimes laser-induced plasma spectroscopy (LIPS)[4].

It is a powerful and versatile analytical technique for elemental analysis, which could be utilized for an enormous range of materials.

a spectroscopic emission source for LIBS application, laser-induced plasma is distinguished from other plasmas (discharge, inductively coupled plasma…) by its violent expansion into the ambient gas.

Taking into account the temporal and spatial evolution of the plasma represents indeed a key point for the LIBS technique.

Spectral features such as emission lines, peak intensity and integrated intensity, etc.

are used for the determination of elemental concentration of the target or discriminate one material, organic or inorganic, from another through their unique spectral signatures.

Laser ablation of Nickel, Copper, Molybdenum, Indium, Tungsten and Gold by short ultraviolet laser pulses (0.5 ps, 248 nm) in vacuum is reported for the first time. For Nickel and Indium, ablation is also studied in air to demonstrate the influence of the ambient atmosphere.

Metal ablation in air is significantly less efficient than in vacuum. Electromagnetic waves (emw) plays an important role in spectroscopy. The behavior of emw is described by Maxwell's equations [5]. Light, laser, x-rays and gamma rays are electromagnetic waves used in a wide variety of spectral applications. Radio and laser emw are used in mineral exploration and determination of the concentration of trace elements [6]. Laser is used also in medicine in surgery and curing some diseases, beside other biological applications..

1.2Research Problem:

Water is very important for human body. Therefore any poisonous chemicals affect the health directly. Thus it is necessary to reduce or get rid of these chemicals. No intensive research in Sudan is concerned with minimization or getting rid of the toxic chemicals has been done.

1.3 Aim The Work:

The aim of this work is to see how adding the ordinary black or green tee affect the water constitunts. The work aims also to determine the elemental composition of the black and green tea

1.4 Literature Review:

Many attempts were made to use spectral techniques to determine the elemental composition os different samples. In the paper of Mohammed Ali Haimid he study the change of the blood exposed to He - N laser. He used the Fourier transform infra-red and ultra violet spectrometers and found that some of the chemical bonds changes or broken due to the laser beam exposure [7]. The work done by Sadeq Ameen for some Yamen rocks utilized induced laser coupled plasma, the laser spectrometer successfully determine the elemental composition and concentration of valuable minerals [8]. Fadhl Ahmed used the paltinest photometer to determine the elemental composition and concentration of trace elements in water successfully [9].

CHAPTER TWO

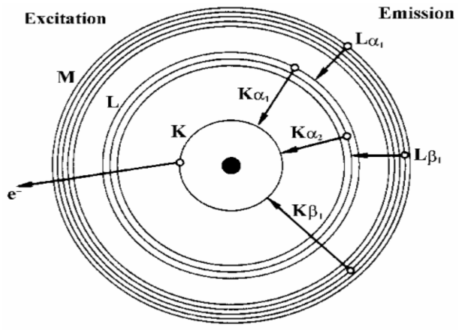
Theoretical background

2.1.Introduction:

The structure of atoms and the nature of atomic spectra beside Induced plasma are presented.

2.2 Atomic Physics:

Using the Planck and Einstein theories , Bohr made general assumptions regarding the structure of the atom.



fag. 4 Bohr's atomic model, shell model

At this time we must emphasize two important points about the Bohr model:

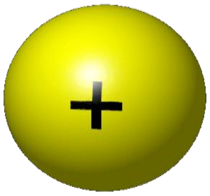
According to his version, electrons revolve around the nucleus, such that the orbit consists of correct number of electron waves. This leads to momentum quantization. According to this:

1. The model correctly fits the quantized energy levels of the hydrogen atom as in ferred from its emission spectra.

2. As the electron is brought closer to the nucleus, energy is released from the system.

The nature of electrons had been elucidated by the experiments of Thomson and Millikan, but the nature of the positive particles was entirely unknown. scientists had exposed that atoms contain electrons and positively charged particles. The definitive experiment that showed how charges and masses are distributed in an atom was carried out in 1909 by Ernest Rutherford. with negative electrons embedded in the positive "dough" of the atom in a way that balanced repulsion between like chargesand attraction between unlike charges. Rutherford tested this model of the atom by using subatomic projectiles to bombard atom.

a-Protons

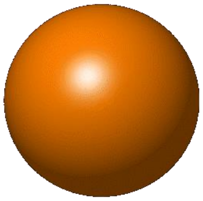


Fag.6 Protons are positively charged particles found inside the nucleus of an atom

Protons are positively charged particles found inside the nucleus of an atom. Each element has a unique atomic number (a unique number of protons).

This leads to the discovery of protons and neutrons. The number of protons and neutrons are equal in most cases.

b-Neutrons

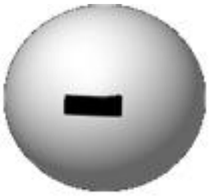


Fag . 7 Neutrons are the other particle found in the nucleus of an atom

Neutrons are the other particle found in the nucleus of an atom. Unlike protons and electrons, however, neutrons carry no electrical charge and are thus "neutral."

Atoms of a given element do not always contain the same number of neutrons.

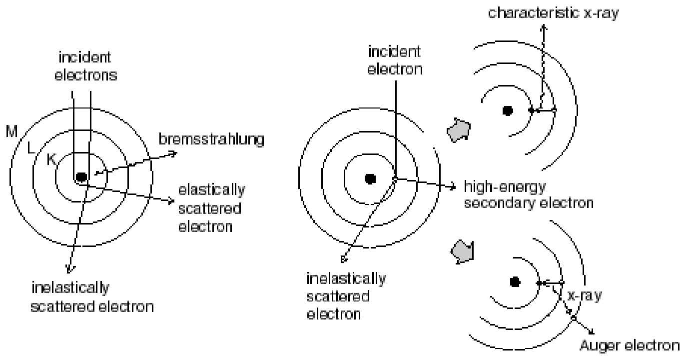
c-Electrons



Fag . 8 Electrons are negatively charged particles that surround the nucleus

Electrons are negatively charged particles that surround the nucleus in “orbits” similar to moons orbiting a planet.

The sharing or exchange of electrons between atoms forms chemical bonds, which is how new molecules and compounds are formed The mass of an electron is about 1/2000th of the mass of a proton or a neneutron.Atom is the basic structure from which all matter has been formed. The electrons which travel around the nucleus of an atom are confined to discrete shells. The electrons in a given shell are bound to the nucleus such that it requires a fixed amount of energy to free the electron from the nucleus. This amount of energy is referred to as the binding energy. Each shell is designated by a letter beginning with “K” assigned to the shell closest to the nucleus and continuing with “L”, “M”, “N” etc as the shells get larger or farther from the nucleus. Thus the “K” shell is the most tightly bound to the nucleus and consequently has the largest binding energy **.**

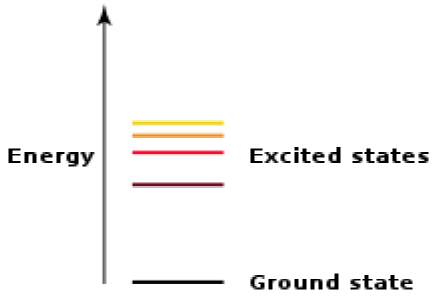


Fag . 11 **Excitation, Ionization, X-ray production**

“K” being the innermost or smallest shell is designated as n=1 with “L” being designated as n=2 etc. The number of electrons that can exist in each shell is specified by the simple equation $2n^2$ where n represents the number of the shell Initially the shells are filled in order so that as the atomic number (Z) increases, the electrons appear in the lowest shell that can accommodate them. For example, potassium has 19 protons and 19 electrons. From the table above it would appear that potassium should have 2 electrons in the K shell, 8 in the L shell and 9 in the M shell. However, it actually only has 8 in the M shell and the last electron is in the N shell. As the atomic number increases, eventually, the M shell is filled with its full complement of 18.

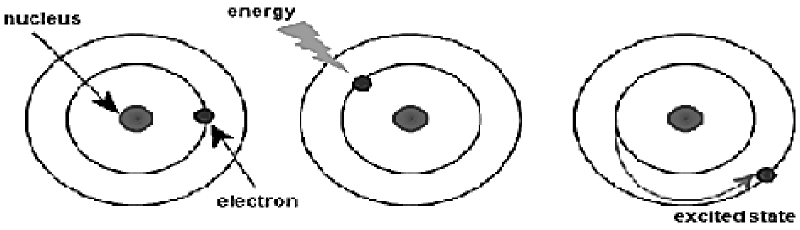
The binding energy for the K shell of hydrogen is - 13.5 eV Similarly for tungsten, the K As can be seen from the table ,and the binding energy decreases as the shells become larger, decreasing to about - 0.5 eV for the O shell. Since the binding energies represent the lack of freedom possessed by the electrons, they are designated here as negative energies where the higher the negative number the more tightly bound.

Excitation is an elevation in energy level above an arbitrary baseline energy state. In physics there is a specific technical definition for energy level which is often associated with an atom being raised to an excited state.The temperature of a group of particles is indicative of the level of excitation . In quantum mechanics, an excited state of a system (such as an atom, molecule or nucleus) is any quantum state of the system that has a higher energy than the ground state (that is, more energy than the absolute minimum).



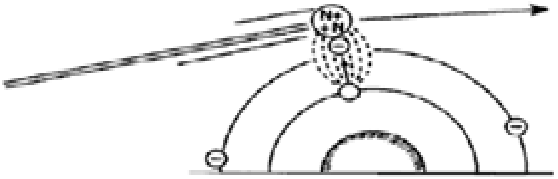
Fag . 12 This is termed excitation

The electron is considered “excited” because it occupies an orbit which is less tightly bound and it left a vacancy in a more tightly bound orbit. It possesses excess energy compared to where it should be.



Fag . 14 The energy needed to raise an electron

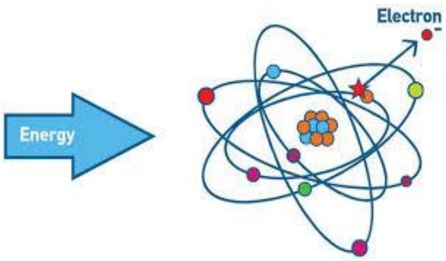
The energy needed to raise an electron to a higher orbit can originate from many sources such as charged particle interactions, photon interactions or particle collisions .



Fag . 15 Simple electrostatic attraction or repulsion

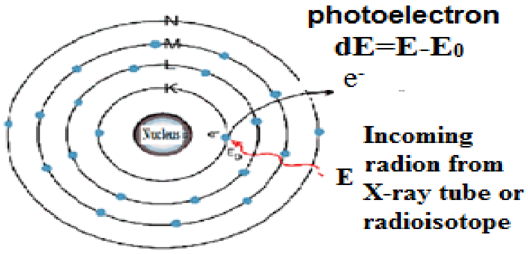
A charged particle does not have to attack the electron to impart energy. Simple electrostatic attraction or repulsion (depending on whether the particle is positively or negatively charged) can be sufficient to induce the electron to change its orbit provided enough energy is transferred (IAEA, 2002).

The atomic collision process is in general, a complex phenomenon and the interaction results in processes like elastic scattering, which dominates at low collision energies, excitation processes to bound and continuum states (ionization). electron exchange processes and correlation phenomenon of coherent impact as identified in the coincidence measurements. We are going to investigate first the ionization of atomic gases by electron impact. The ionization of atoms by impact processes is of importance in a number of feints such as plasma physics, radiation physics, atmospheric physics, astrophysics and fusion physics (Hans, 2010). There is an obvious advantage in obtaining both the positive ion and the negative ion chemical ionization mass spectra Over the past 30 years, negative ion chemical ionization has become a powerful analytical method. In many cases it offers increased sensitivity and selectivity and frequently it provides information which is complementary to positive ion chemical ionization. (Harrison, 1992).



Fag . 16 Ionization of an outer shell electron

The electron ejected from the atom need not be one from the highest orbit or shell. In order to ionize such an electron, the incoming radiation must possess an amount of energy at least equal to the binding energy of the electron. Even the most tightly bound electrons, those in the K-shell can be ejected during the ionization process.



Fag . 17 Ionization of a K- shell electron

If the incoming radiation possessed only an amount of energy equal to the binding energy of the electron, the electron would be freed from the atom but would have no surfeit kinetic energy to travel away from the atom **(IAEA, 2002).** The incoming radiation possesses energy greater than the binding energy of the electron, then the excess energy is merely the initial energy of the incoming radiation (E) minus the energy expended to overcome the binding energy of the electron (E_o). This excess energy DE is given to the freed electron as kinetic energy which permits it to travel far from the atom from which it originated. This will ultimately be the source of radiation dose which will be discussed in future modules. The energy of a photon is proportional to its frequency: $E = hf = hc/l$, where h is Planck's constant = 6.63×10^{34} j·s, f is the frequency of the photon (s^{-1}), c is the speed of light and l is the wavelength of the photon.

2.3 Atomic Spectra:

Atoms emit characteristic radiation due to the electronic transition between energy states [10].

A- Emission Spectroscopy:

Light emit electromagnetic radiation of different wavelengths when the elements or their compounds are heated either on a flam or by an electric field they emit energy .The emitted wave length depends on the form of the composition of material since it different of each element of periodic table. The emission spectrum characteristics of some elements are plainly visible to the naked eye when these elements are heated. These definite characteristics allow elements to be identified by their atomic emission spectrum .not all emitted lights are perceptible to the naked eye as spectrum also includes ultraviolet ray and infrared lighting .an emission is formed when an excited gas is viewed directly through a spectroscopy Emission spectroscopy is technique which examines the wavelength of photons emitted by atoms or molecules during their transition from an excited state to a lower energy state. Each element emits a characteristic set of discrete wavelengths according to its electronic structure, and by observing these wavelengths the element composition of the sample can be determined (Carroll & Bradleyw,2007)

B- Absorption Spectroscopy:

Refers to spectroscopic techniques that measure the absorption of radiation, as a function of frequency or wavelength, due to its interaction with a sample. The intensity of the absorption varies as a function of frequency and this variation is the absorption spectrum. Absorption spectroscopy is performed across the electromagnetic spectrum. Absorption spectroscopy is employed as an analytical chemistry tool to determine the presence of particular substance in a sample and in many cases to quantify the amount of the substance present. Infrared and ultraviolet- visible spectroscopy are particularly common in analytical applications **(David,2002)**

2.4 Laser spectroscopy:

Laser is a very intense light having single frequency with coherent waves. It results from the process of stimulated emission of radiation.

A variety of configurations and methods for laser spectroscopy have been developed. Infrared and far-infrared spectroscopy are commonly used for gas analysis and identification of chemical structures while visible and ultraviolet spectroscopy are extensively used for quantitative analysis of atoms, ions, and chemical species in solution. Many technical literatures describe these spectroscopic methods and their application. We will consider a few selected types of laser spectroscopy as examples. The high mono chromaticity of laser light, ideally, makes it possible to induce transitions between exactly one initial and one final vibration-rotation level in the initial and final electronic states, respectively. For any subsequent process this reduces the large number of intermediate states which otherwise might participate in a second step absorption or emission process.

Thus, the complexity of the spectra is dramatically reduced and precision is substantialy improved [10].

A- Laser absorption spectroscopy:

Laser-based absorption spectroscopy (AS) is a powerful technique for qualitative and quantitative studies of atoms and molecules. An important field of use of AS is the detection of species in trace concentrations, which has applications not only in physics and chemistry but also in biology and medicine, encompassing environmental monitoring, regulation of industrial processes and breath analysis. Although a large number of molecular species can successfully be detected with established AS techniques, there are some applications that require higher sensitivity, selectivity and accuracy, yet robust and compact instrumentation .In atomic absorption spectrometry (AAS), both with the classical flame AAS and with furnace AAS innovation took place. Remarkable efforts, however, were made to use all types of methods allowing volatile species generation with metals.

B- Laser emission spectroscopy:

As soon as the laser was developed in the early 1960s, spectra chemists began investigating its potential uses. An early observation was that a pulsed laser could produce a small plasma in air. The emission from that plasma from 1960 onwards, increasing availability of intense, monochromatic laser sources provided a tremendous impetus to a wide range of spectroscopic investigations .From 1960 to 1980 the analytical capability was so inferior to that of the conventional spark and laser technology was in its infancy, so that the technique was less favored than a related one – laser ablation into a conventional plasma source. Here the laser was used to vaporize a small amount of sample for analysis by, for example, the conventional electrode spark. However, that was not the only way the laser could be used in spectra chemistry. The development of tunable dye lasers meant that one could illuminate a prepare source of atoms with radiation resonant with a transition in one of the atomic species. Then either the absorption of the laser beam or the laser-induced fluorescence could be used as an analytical signal. These techniques discriminated against background and increased the signal to noise considerably by recycling the same atoms many times. Sometimes the atoms were placed in the laser cavity itself [11].

2.5 Laser Induced Breakdown Spectroscopy:

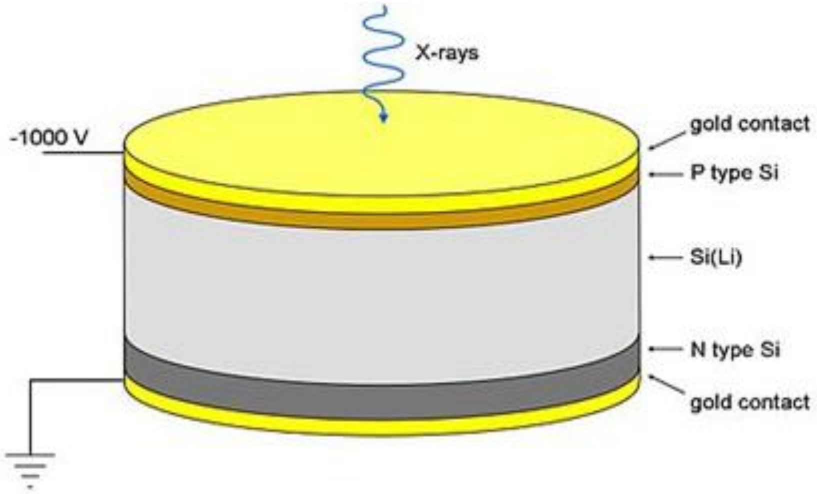
Laser-induced Breakdown Spectroscopy (LIBS) is a novel method of elemental analysis based on a laser-generated plasma. Pulses from a laser are focused on a sample to atomize a small amount of material resulting in the formation of a microplasma. Because of the high plasma temperature, the resulting atoms are electronically excited to emit light. The plasma light is spectrally resolved and detected to determine the elemental composition of the sample based on the unique emission spectrum of each element. Because of the simplicity of the method, it is suited for analyses that cannot be carried out using conventional methods of atomic emission spectroscopy (AES) **(Cremers & Multari, and Knight, 2006)** Laser Induced Breakdown Spectroscopy (LIBS) diagnostics is currently used in many diagnostic applications, being a candidate either to substitute or to be complementary to Inductively Coupled Plasma (ICP) for some applications, e.g. analysis of solid samples .The technique is based on the interaction of a laser with a solid surface generating a plasma plume whose emissivity is analyzed to obtain information on the concentrations of relevant species. LIBS therefore is a diagnostic technique based on Optical Emission Spectroscopy (OES) and as such is similar to any emission spectroscopy including ICP. A major advantage of LIBS over ICP is the extreme simplicity in sample preparation, while both techniques share the same underlying assumption that the formed plasma, due to its high ionization degree, is in a state of Local Thermodynamic Equilibrium (LTE), implying that the Boltzmann plot method can be used to determine the plasma temperature. In LTE, one can also obtain absolute values of the concentration of the different species by measuring the emissivity of a transition. **(Sergio, 2014)** In recent years the application of the laserinduced breakdown spectroscopy (LIBS) technique w1x to 'real life' problems have greatly grown in number and in variety. The feasibility of rapid multi-elemental analysis and the lack of a direct contact with the sample are appealing prop-erties that conquer more and more new LIBS users. In fact, the analytical performances of the LIBS technique depend strongly on the choice of experimental conditions, which have to be carefully studied in each particular application. Main parameters affecting the performance are the laser wavelength, the pulse energy, the time interval of observation and the geometrical set-up of the collecting optics **(Tognoni& Palleschi& and Cristoforetti, 2002).**

2.6 ICP-OES Principle:

Laser-induced breakdown spectroscopy (LIBS), also called laser-induced plasma spectroscopy, laser spark spectroscopy, and laser optical emission spectroscopy, is a multi-elemental analytical technique based on atomic emission spectroscopy (AES). It employs a high-energy laser pulse as the vaporization, atomization, and excitation source to create a high-temperature microplasma at the surface of the target . Firstly, the process is initiated by reflection or absorption of energy by the sample from the pulsed laser. Then, the absorbed energy is rapidly converted into heat, resulting in melting and vaporization of small portion of material into ionized gas when the temperature approaches the boiling point of the material. The removal of particulate matter from the surface leads to the formation of a vapour in front of the sample. When this vapour condenses as droplets of submicrometer size, it leads to scattering and absorption of the laser radiation inducing heating, ionization and plasma formation **(El-Deftar, 2014)**. A pulsed laser beam is focused onto The surface of a substance to be analyzed the radiation. energy is locally coupled into the material and the material starts to evaporate .within this material vapor and the surrounding gas atmosphere a plasma is generated leading to the excitation of material constituents and their spontaneous emission of radiation. The plasma decays and emits element specific radiation. This emission is resolved spectrally and is detected by spectrometer. For solid substances a crater is formed finally the evaporated material is removed partially from the interaction zone driven by the intrinsic dynamics of the plasma expansion **(Edrees R. A., 2017).**

The isotopic excitation source $^{109}_{48}$Cd is used only to measurer the standard G308 and compare its analysis with tube excitation system. This isotopic source ($^{109}_{48}$Cd) is an annular source of an initial nominal activity of 25 mCi. In this type of decay the $^{109}_{48}$Cd is decays to Ag by electron capture transformation and produces characteristic X-rays of the daughter isotope $^{109}_{47}$Ag 19 - In the isotopic Cd vacancies are created in the K-shell, electrons fall from L, M and N shells to fill the created vacancies. In the process K, and K characteristic X-rays of the daughter isotope $^{109}_{47}$Ag are emitted of energies 22.1 keV (for K,) and 24.9 keV (for KB), respectively. The average energy that leads to excitation of elements in the presented sample is taken to be 22.6 keV. **(Ahmed, 2015)**

^{241}Am isotopic source is produced as a daughter in the decay of the isotope ^{241}Pu by β transformation. ^{241} Am decays by emission of y-rays of energy E,= 59.5keV. These gamma-rays are used to excite elements in XRF analysis. **(Marghani, 2013).**



XRF setup with Si(Li) Detector

2.7 Spectroscopy:

Spectroscopy is study of the interaction of electromagnetic radiation with matter.When matter is energized (excited) by the application of thermal electrical nuclear or radiant energy electromagnetic radiation is often emitted as the matter relaxes back to original (ground) state. The spectrum of radiation emitted by a substance that has absorbed energy is called an emission spectrum and the science is appropriately called emission spectroscopy **(Edrees R. A., 2017)**

2.7.1.Types of spectroscopy:

Atomic spectroscopy:

The study of transitions, in absorption or emission between electronic states of an atom, is atomic spectroscopy. It deals with the interaction of electromagnetic radiation with atoms which are most commonly in their lowest energy state. Atomic spectra involve only transitions of electrons from one electronic energy level to another .The atomic spectroscopy categorized into two types **(Larkins & Payling, 2000)**

2.7.2- ICP-OES:

The spectroscopy in optical emission spectroscopy from reached toms in inductively besplel plastia This technique is an as inductively coupled per optical mission spectroscopy has number of was over absceptica spec were in the previsto cheaper in lading the atelity to mesure 75 elements With LICP-OES, there are a substantial amount of elements I conako he analysed simultanely, inel of one-by-one with AAS. Also, since LICP-OES is an emissionbased cek mas memen, here is no need for all of the lamps lle are spiral with atomic absorption spectroscopy **(Hma Athui, 2016)** . Te majority of the vinegars were in glass bottles; however, several were in plastic bottles. Sample containers were (500 ml) and an identification code was assigned for each sample: WV for wine vinegar, ACV for apple cider vinegar, RV for rice vinegar, SCV for sour-cherry vinegar, DV for date vinegar and BV for balsamic vinegar. Te samples were stored at 4±1ºC prior to analysis. Sample selection was completed in two stages: one sample of each product was obtained from the supermarkets; and afer one month, a second visit was made to the same supermarkets to obtain a second sample. Tis procedure was utilized to include different batches of each product. Te numbers of samples of each product depended on the numbers of brands available in the market. **(Turkey, 2010)**

1- The ICP Discharge:

The inductively coupled plasma discharge used today for optical emission spectrometry is very much the same in appearance as the one described by Velmer Fassel in the early 1970's. Argon gas is directed through a torch consisting of three concentric tubes made of quartz or some other suitable material, A copper coil, called the load coil, surrounds the top end of the torch and is connected to a radio frequency (RF) generator. **(Charles& Fredeen, 1997).**

2- Detection of Emission:

LICP-OES, the light emitted by the excited atoms and ions in the plasma is measured to obtain information about the sample. Because the excited species in the plasma emit light at several different wavelengths, the emission from the plasma is polychromatic. This polychromatic radiation must be separated into individual wavelengths so the emission from each excited species can be identified and its intensity can be measured without interference from emission at other wavelengths. The separation of light according to wavelength is generally done using a monochromator, which is used to measure light at one wavelength at a time, or a polychromator. **(Charles& Fredeen, 1997).**

3- Extraction of Information:

Extracting qualitative and quantitative information about a sample using LICP-OES is generally straightforward. Obtaining qualitative information, i.e., what elements are present in the sample, involves identifying the presence of emission at the wavelengths characteristic of the elements of interest. In general, at least three spectral lines of the element are examined to be sure that the observed emission can be indeed classified as that belonging to the element of interest **(Charles & Fredeen, 1997).**

4- Performance Characteristics:

LICP-OES technique is applicable to the determination of a large number of elements. The detection limits for these elements are generally in the mg/L (ppb) range. As in many techniques, the detection limit is regarded as the lowest concentration at which the analyst can be relatively certain that Calibration curve used for LICP-OES. element is present in a sample. Measurements made at or near the detection limit, however, are not considered to be quantitative. For purposes of rough quantitation (±10%), it is recommended that an element's concentration should be at least five times higher than the detection limit.

2.8 Applications of ICP-OES:

[1]Environmental Applications:

To meet increasingly tough regulatory and budgetary challenges, environmental laboratories must deliver accurate results faster, more reliably, and more cost-effectively than ever before.

[2]Food & Agriculture Applications:

Accurately quantifying trace metals is not only vital to food safety but can also identify fraudulent mislabeling of a food’s origin, as the metal content can be used to determine provenance. Quantifying major and minor elements in soils, crops and fertilizers is an important diagnostic agronomy tool.

[3]Metallurgical Samples ICP-OES:

Appropriately in this case . Metallurgical Samples LICP-OES is used widely together with X-ray fluorescence and other techniques for the determination of major, minor, and trace elements in metals and other metallurgies laser ablation, arcs and sparks, as well as glow discharge techniques can be used most . Some representative applications of the LICP-OES tech- unique for metallurgical materials include the determination of minor and trace elements in alloys, high precision determination of Si in steels, determination of contaminants in high-purity aluminum, analysis of superconducting materials for trace contaminants, and lanthanide elements determination in manganese nodules. **(Cazes, 2007)**

CHAPTER THREE

Experimental Work

3.1 Introduction:

In this work induced coupled Plasma (ICP) was used to determine the concentration of trace elements in red and green tea beside fresh water. The concentration of trace elements water. The concentration of trace elements in the red and green tea waters were also sound. This chapter is concerned with methods, results, discussion and conclusion.

3.2 Materials and instruments:

5 samples were prepared in this work. Two of them are the red and green tea, the third is a pure water. The red and green tea were mixed separately in water and heated till they reached the boiling point. The ICP spectrometer then was used to determine the concentration of trace elements in the 5 sample.

3.3 Methodology:

The row red tea water was examined first by ICP spectrometer. The fresh pure water was also examined by ICP to determine the trace elements existing in them. One spoon of the red tea was mixed with the fresh water in a metallic container then heated by a heater till it boils. The mixture was left boiling for about minutes. The mixture then left to cool and to allow the red tea waste to precipitate for about minutes. The red tea water was filtered by a filler paper to allow the filtered tea water to be collected in a tea cup. This filtered.

Figure (2): Experimental set up of the heater and tea container and filter red tea water was examined using ICP spectrometer. The same procedures were repeated for a green tea.

3.4 Results:

The following tables exhibit the concentration of trace elements in teas and waters samples.

Table (1): water and red tea trace elements before and after interaction.

Element	pure water C (mg/l)	red tea water C (mg/l)	pure rewired tea before immerse C (mg/l)	pure rewired tea after immerse C (mg/l)
AL		40		
Ca	200	11		8700
K	24	170	310	2400
Mg	86	5		230
Mn	3	2.3		1000
Si	74	0.35	1.7	84
P		28	25	5500
S	28	250	5300	3800
Cu		1.7	29	
B			2.7	12
Na	250		630	290
Nb			6.5	3.3
I	1.1			2300
Sr			10	28

Zn				15
Cd	150			1.3
Fe	4.9			460
Cr				3.9
Li				9.1

Table (2): Water and green tea trace elements consent actions before and after interaction.

Element	pure water C (mg/l)	green tea water C mg/l)	pure row green tea before immerse C (mg/l)	pure row green tea after immerse C (mg/l)
AL			3500	2.900
Ca	200	930	640	640
K	24	140	3400	1200
Mg	86	12	320	260
Mn	3	7.4	1600	1400
Si	74	3.9		7
P		81	7400	5000
S	28	840	12000	2800
Cu	6.2		70	62
B			9.9	7.4
Na	250		360	250
Nb			3.3	3
I		46	2900	2200
Sr	1.1		15	

Zn			24	17
Cd			1.2	1.6
Fe	150		560	460
Cr	4.9		1.7	3.4
Ce		3.7	15	
Li		24		
Nd		1.3	8.8	6.5
OS		2.6		
Pr		3.1		
Sb		4.4		
Sm		1.7		
Th		7.2		9.3
Co			2.3	

3.5 Discussion:

Table (1) shows that the concentration of some elements like Ca, Mg, Si, Na, Sr, Fe and Cr decreases in red tea water. This may be altributed to the fact that some of them may be removed by tea water like Ca, Mg, Mn, Si Sr, Fe and Cr. Other maybe removed from both pure water or row red tea by the filter like Na, S and Cr. New trace elements which does exist in both pure water or row red tea were found. The only observed one is AL which may come from the metallic container or the environment. It is also very interesting to note that, expect AL, all other trace elements concentrations in red tea water are less then the sum of their concentrations in pure water and row red tea.

In view of table (2) for green tea it is clear that the concentrations of Mg, Si, Na, Sr, Fe and Cr in green tea water is less than that for pure water only. The sum of all trace elements in pure water and row green tea, except Ca, is greater than of green tea water. Only few trace elements with very low concentrations like Li, Na, Os, Pr, Sb, Sm,, Th and Co comes from the surrounding it may come from the tube or container used. It may also come from air.

3.6 Conclusion:

The analysis of red and green tea water shows that the filtration process gets vid of many un wanted trace elements. It was also observed that some other trace elements which does not exist in water or row tea material are found in red and green tea water which may come from cups or containers or surrounding medium including air.

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