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Influence of Waste Water Used in Irrigation on the Physical Properties of Olive Oil in Palestine

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This Thesis is Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Physics, Faculty of Graduate Studies, An-Najah National University - Nablus, Palestine.

2015

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Dedication

This thesis is dedicated to my husband Motaz,

to my parents, as well as,

to my kids Ayham and Massa

to my brothers and sisters.

With respect and love.

This thesis is also dedicated to Prof. Issam Rashid Abdelraziq

for his support,

to everyone who helped me,

to everyone who gave me a good support.

Acknowledgement

I'd like to express my sincere appreciation to my supervisors, Prof. Issam Rashid Abdelraziq for his helpful efforts and continual encouragement.

I would like to thank Prof. Sharif Mohammad Musameh who helped me to finish this research and gave me a good support.

الإقرار

أنا الموقعة أدناه مقدمة الرسالة التي تحمل العنوان:

Influence of Waste Water Used in Irrigation on the Physical Properties of Olive Oil in Palestine

اقر بان ما اشتملت عليه هذه الرسالة إنما هو نتاج جهدي الخاص، باستثناء ما تمت الإشارة إليه حيثما ورد، وان هذه الرسالة ككل أو من جزء منها لم يقدم من قبل لنيل أية درجة أو بحث علمي أو بحثي لدى أية مؤسسة تعليمية أو بحثية أخرى.

Declaration

The work provided in this thesis, unless otherwise referenced, is the researcher's own work, and has not been submitted elsewhere for any other degree or qualification.

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List of Symbols and Abbreviations

Amu	Atomic Mass Unit
APCI-MS	Atmospheric Pressure Chemical Ionization Mass Spectrometry
cP	Centipoises
cSt	Centistokes
EHD	Elasto hydrodynamics
FFA	Free Fatty Acid
GFAAS	Graphite Furnace Atomic Absorption Spectrometry
HDL	High Density Lipoproteins
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectrometry
ICP-EOS	Inductively Coupled Plasma Optical Emission System
ICP-MS	Inductive Coupled Plasma Mass Spectrometry
IOOC	International Olive Oil Council
LDL	Low Density Lipoproteins
NMR	Nuclear Magnetic Resonance
Pa.s	Pascal – Second
PPm	Part Per Million
PPt	Part Per Trillion
PTDI	Provisional Tolerable Daily Intake
PVC	Pressure Viscosity Coefficient
RPM	Revolution Per Minute
RWW	Reclaimed Waste Water
η	Dynamic Viscosity or Absolute Viscosity
ν	Kinematic Viscosity
ρ	Mass Density of Liquid
γ	Shear Rate
τ	Shear Stress
S_{1a}	Misseli and Hawara samples with trees depend on rain water of crop 2013
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Abstract

In this study, olive oil samples of different irrigation system in Palestine were studied. The density, refractive index, acidity and viscosity of the samples were measured. The results indicate that acidity increase for samples with trees irrigated by waste water which can be classified as Lampante oil. The experimental results of viscosity showed that the viscosity increased for all samples were irrigated by waste water.

The concentration of some metals in olive oil samples irrigated by waste water have high concentration compared with literature. These metals are: calcium, sodium, potassium, magnesium and zinc. The concentrations of iron, copper and lead for irrigated samples by waste water are greater than concentrations of established by standards of IOOC. The presence of these metals in olive oil may be due to the abundance of them in waste water. The concentration of cadmium and manganese found at high concentration for Hawara samples because it is industrial area.

Chapter One

Introduction

1.1 Olive Trees

Olive trees are one of the perennial trees which were planted by early humans beside trees of date like grapes and figs since early Bronze age (Liphschitz *et al*, 1991 and Zohary *et al*, 1975). Olives home in West Asian countries which is also known as Mediterranean Countries and that's due to middle environmental conditions (Kailis *et al*, 2007).

Palestine is one of the West Asian Countries which planted olive trees long time ago. The total agricultural land cultivated with olive trees forms 80% of the land which is approximately 93000 hectors, which means more than ten millions of olive trees have been planted (Qutub *et al*, 2010).

Approximately Palestinian 10000 families have been relying on olive harvest for their livelihoods. Olive is considered as one of the main crops in Palestine, it is represented as the symbol of Palestinian stead fastness. On the other hand, olive groves have formed the life and Identity of the Mediterranean's (Loumou *et al*, 2003).

The lack of care for olive trees and its rips causes the loss of sensual properties of olive oil, which is classified to be not virgin (http://go.world bank.org/ 2013). Olive trees quality depends on the conditions in which they live, mostly grown in arid, semi-arid regions and difficult climatic condition, olives need low temperature in winter and a warm dry climate in the summer (Kailis *et al*, 2007).

Olive rips have many components such as water 50.0%, oil 22.0%, proteins 1.6%, carbohydrate 19.0%, cellulose 5.8%, minerals 1.5% for instance (copper, iron, lead), sugar at low contents 2.6 – 6.0% and contains 1 - 3% hydrophilic component which they are phenolic acid, phenolic alcohol, flavonids, secoiridoids (Gharsallaoui *et al*, 2013; Ghanbari *et al*, 2012; Gunstone, 2002 and Segal *et al*, 2011).

There are different varieties of olive drupe in different regions which differ in size, color, oil content, fatty acid composition and other properties.

1.2 Olive Oil

Olive oil is an edible oil, which is known as one of the components of Mediterranean diet along with fruits and vegetables. Olive production is spreading over worldwide (Owen *et al*, 2000 and Zeiner *et al*, 2005).

There are many countries that are co - producers of virgin olive oil like Spain, Italy, Greece, Syria, Tunisia, Turkey, Morocco and Palestine. Olive oils intake is greatly growing for its known health effect, correlated with the reduction incidence of coronary heart disease, and that’s due to its natural antioxidant components such as hydrophilic phenols, which plays an essential role in reducing the formalization of toxic compound such as lipids peroxides (Andrewes *et al*, 2003; Rahmani *et al*, 1991 and Servili *et al*, 2002).

Olive oil is used to lower the possibility of having certain kind of cancer, such as colon disease, and are used in making different kinds of ointments (Benincasa *et al*, 2007; Visioli *et al*, 1995 and Zeiner *et al*, 2005).

Olive oil represents a powerful source of vitamin E, polyphone's and mono-unsaturated fatty acid, with positive result to increase high - density lipoproteins (HDL) and reduce low - density lipoproteins (LDL)(Al Jamal *et al*, 2011; Cabrera - Vique *et al*, 2012 and Roussel *et al*, 2002). Olive oil represents the main source of fat in diets (Loumou *et al*, 2003 and Visioli *et al*, 1994).

The color of virgin Olive oil is attributable to chlorophylls and carotenoids content, which is connected to green pigment and duration storage.

Olive oil has been distinguished from other oils due to the presence of its volatile elements, which rises during the extraction of oil and becomes less during the oil storage. The presence of volatile elements is responsible for giving oil a distinctive flavor related to sensory properties and quality (Kalua *et al*, 2007).

The essential volatile flavor compounds of olive oil are Hexanol and 1 – Hexanol which are created in maximum concentration due to oxidation of fatty acid (Kalua *et al*, 2007). Olive oil storage decreases other volatile compounds such as Aldehydes and esters (Kiritsakis, 1998).

Olive oil quality is affected by many factors such as climate, nature of water used for irrigation of olive trees, fertilizing applications, processing equipment, period of repining their fruit and other factors (Alsaed *et al*, 2011 and Garcia *et al*, 1996).

Crude oil has been manufactured without cleaning, such as extra virgin olive oil, and could contain high quantities of trace elements, which are found in elements at low concentration ($\frac{mg}{Kg}$ or less) in agro ecosystem (Cabrera - Vique *et al*, 2012).

Olive oil should be stored and kept in dark places, at temperatures lower than 25℃.

The International olive oil council (IOOC) classified the olive oil into positive and negative features according to some sensory properties, such as production method, chemical design and flavor (http: // international olive oil .org/ 2014).

1.3 Previous Studies

1.3.1 Trace Elements

Trace elements are found in the natural environment, some are hazardous to organisms and others are used in life (Adriano *et al*, 2001). Some of the trace elements include copper (Cu), zinc (Zn), manganese (Mn) and iron (Fe) which are essential for plant growth (Zheuli *et al*, 2005). These metals are known as heavy metals because they have a density greater than 0.5 gm/cm^3 and they are described as toxic when they are found at high concentration greater than 0.5 gm/cm^3 (Adriano *et al*, 2001).

Trace elements, such as cobalt (Co) and selenium (Se) are not essential to the plant growth but they are required for animals and human beings. Other trace elements are essential for humans nutrition which are sodium (Na), potassium (K), calcium (Ca), iron (Fe), magnesium (Mg), copper (Cu) and zinc (Zn) (Zeiner *et al*, 2005).

Other trace elements like cadmium (Cd), lead (Pb), chromium (Cr), nickel (Ni), mercury (Hg) and arsenic (As) have the toxic effect for all living organisms even if they are used in small quantities and stored at low concentration for long time periods (Pehlivan *et al*, 2008 and Zheuli *et al*, 2005).

Many articles have been published to describe the relevance between cancer disease and specific trace elements such as cadmium (Cd), selenium (Se), arsenic (As) and other elements which are toxic (Sensil *et al*, 1999).

Cindric and Michaela describe the presence of trace elements during their study on different oil types (olive, rice oil, sun flower, sesame, hazelnut, grape, soya) by using microwave assisted digestion followed by inductively coupled plasma atomic emission spectrometry (ICP - AES). This device measures the presence of trace elements with high sensitivity. It is regarded as best method for complicated analysis which determines the content. It does not require a large quantity of oil samples. However this technique loses volatile metals from oil samples (Cindric *et al*, 2007 and Pehlivan *et al*, 2008).

Matos determined the presence of Cu and Ni in vegetable oil using graphite furnace atomic absorption spectrometry (GFASS) (Matos *et al*, 2006).

Cindric et al have studied the presence of Ca, Fe, Mg, Na, and Zn using ICP- AES and measured Al, Cu, Co, Cr, K, Ni, Mn and Pb by using GFAAS method (Cindric *et al*, 2007; Cindric *et al*, 2008).

Trace elements may be analyzed by using inductively coupled plasma-optical emission (ICP - EOS) systems which dominates the inorganic analysis landscape (Marin *et al*, 2011).

Other trace elements such as Ca, Fe, Mg, Na and Zn found in olive oil, have been studied by ICP – AES technique, which was used by Zeiner in a pilot study on the geographical characterization and found the concentration of trace elements (Zeiner *et al*, 2005).

Trace elements which are found in different types of oil due to the influence of human health and nutrition, transports from agro eco system to the environment (Zhenli *et al*, 2005).

The quality of edible oils can be estimated by determine the concentration of several trace elements (Ashraf, 2014).

A group of researchers studied trace elements in olive oil and found the concentration of metals differ as listed in table (1.1)

Table(1.1): The measured results for the concentration of metals, which they were found in olive oil samples with literature values (mg/kg)

Elements	Symbols	Literature Values (mg/Kg)	References
Calcium	Ca	1.25 - 9.01, 2.0 - 2.2	Zeiner *et al*, 2005 and Cindric *et al*, 2007.
Iron	Fe	14.36 - 118.55, 14.2 - 16.6, 12.5 - 139.0, 13.10 - 18.46, 16.2 - 45.3, 25.3 – 273	Sahan *et al*, 2004; Cindric *et al*, 2007; Bencase *et al*, 2007; Zeiner *et al*, 2005; Zhu *et al*, 2011; Vique *et al*, 2012.

Copper	Cu	0.248 - 0.276, 0.03 - 0.05, 0.04 - 4.51, 3.35 - 18.27, 0.73 - 2.55, 3.50 - 6.67, 2.17 - 3.80	Zhu *et al*, 2001; Cindric *et al*, 2007; Zeiner *et al*, 2005; Vique *et al*, 2012; Sahan *et al*, 2007; Biricik *et al*, 2006 and Acar, 2012.
Zinc	Zn	1.24 - 1.58, 3.1 - 3.7, 2.28 - 4.03, 2.19 - 11.53, 10.58 ± 2.01, 2.33 - 4.44	Zhu *et al*, 2011; Cindric *et al*, 2007; Zeiner *et al*, 2005; Sahan *et al*, 2004; Sahan *et al*, 2007 and Acar, 2012.
Cadmium	Cd	2.39 - 2.78, 3.2 - 8.1, 0.06, 0.04 - 0.11	Zhu *et al*, 2011; Zeina *et al*, 1997; Madejan *et al*, 2006; Acar, 2012.
Nickel	Ni	0.046 - 0.061, 1.1 - 1.9, 0.07 - 2.26, 10.2 - 18.32	Zhu *et al*, 2011; Cindric *et al*, 2007; Ziner *et al*, 2005 and Vique, 2012.
Lead	Pb	0.009 - 0.018, 0.71 - 0.75, 0.79 - 3.68, < 0.1	Zhu *et al*, 2011; Sahan *et al*, 2007; Cindric *et al*, 2007; Sahan *et al*, 2014
Arsenic	As	0.008 - 0.015, < 0.1	Zhu *et al*, 2011 and Zeiner *et al*, 2005.
Magnesium	Mg	3.2 - 3.8, 2.91 - 3.62, 0.113 - 0.556	Cindric *et al*, 2007; Zeiner *et al*, 2005 and Zhu *et al*, 2011.
Sodium	Na	34.0 - 34.5, 28.77 - 38.03	Cindric *et al*, 2007 and Zeiner *et al*, 2005.
Aluminum	Al	0.10 - 015, 0.08 - 1.11	Cindric *et al*, 2007 and Zeiner *et al*, 2005.

Cobalt	Co	1.12 - 1.15, 0 - 5.45	Cindric *et al*, 2007 and Zeiner *et al*, 2005.
Manganese	Mn	0.009 - 0.200, 0.008 - 0.13, 11.61 - 69.42	Cindric *et al*, 2007; Zeiner *et al*, 2005 and Vique *et al*, 2012.
Chromium	Cr	6.84 - 59.36, < 0.001	Vique *et al*, 2012 and Cindric *et al*, 2007.
Potassium	K	0.05 - 0.19	Zeiner *et al*, 2005.

Levels of trace elements like Cu, Zn, Fe, Mn and Ni cause an increase in the rate of oil oxidation. Where As, Cd and Pb form of other trace elements which are very essential on account of oil toxicity (Zhu *et al*, 2011).

1.3.2 Irrigation System for Olive Trees by Using Waste Water

Olive trees are considered relatively tolerant to salinity; they are defined as a crop moderately tolerant to salinity. Olive isn't eaten fresh and only wasted after processing, so this reduces the exposure to pathogenic and microorganisms which are found in reclaimed waste water (RWW) (Segal *et al*, 2011 and AL-Shdiefat *et al*, 2009). Clean water is better to use for irrigating other crops which are more sensitive to water than olive trees (Gharsallaoui *et al*, 2013).

Recently it has been reclaimed that fresh water is replaced by wastewater because it represents the only water resource that has been used in poor communities (Mass *et al*, 1990 and Chartzoulakis *et al*, 2002). Other countries like Cyprus, France, Italy, Jordan and Tunisia are the only Mediterranean countries that have established national guidelines for the uses of reclaimed wastewater (Angelakis *et al*, 1999).

Reclaimed waste water has been used to increase water resource, protect coastal areas, in addition to nature conservation policies, urbanization and improve living condition (Gharsallaoui *et al*, 2013). The most important use of RWW is to increase the level of certain antioxidant components such as polyphenols (phenolic compounds) which are found in extra virgin olive oil as well as increasing vitamin E (AL - Shdiefat *et al*, 2009; Inglese *et al*, 1996 and Wiseman *et al*, 1996).

Waste water has been regarded as cheaper for irrigation and characterized by higher salinity compared to fresh water (AL - Shdiefat *et al*, 2009).

Many reports described the harmless effect that trace element have on oil when the trees were irrigated with waste water. This is because of the presence of high concentrations of micro organisms such as bacteria, fungi, viruses, helminthes and heavy metals which are found in wastewater such as arsenic, copper, cadmium, lead, chromium, nickel, mercury and zinc (Akpor *et al*, 2010; Gharsallaoui *et al*, 2013 and Tchobanoglous *et al*, 2002).

Bedbabis has noticed high concentration of heavy metals Zn and Mn only after the second year of irrigation, during his study the effect of irrigation with treated wastewater on olive trees grown in arid region in Tunis (Bedbabis *et al*, 2010).

Segal and his group reported that the quantity of nitrate and chloride decreases when olive trees are irrigated with recommended waste water. Segal studied the effect of reclaimed water that is used for irrigation on the growth of olive trees. He found that the concentration of some metals in each of the reclaimed water and pure water differs and the highest concentration of these metals are observed in reclaimed water as shown in table (1.2) which means there is a relation between trace element and irrigation system (Benincasa *et al*, 2012 and Segal e*t al*, 2011).

Table (1.2): Comparing the concentration of metals which were found in each of reclaimed water and pure water in mg/L according to Segal, 2011 and his group studies

Metal	Symbol	Reclaimed water	Pure water
Potassium	K	29.6	4.4
Phosphorus	P	5.8	0
Magnesium	Mg	39.0	28.0
Boron	B	0.2	0.1
Chloride	Cl	323.0	168.0
Sodium	Na	198.0	81.0

A group of researchers compared the effect of irrigation system between fresh and saline water and they found that the amount of total phenols component decrease when irrigated by saline water while vitamin E increases (Ghanbari *et al*, 2012). Al - Absi found that peroxide values were not affected with any irrigation system (Al - Absi, 2008).

1.3.3 Acidity of Olive Oil

Researchers have described various processing systems on olive oil quality, equipped pressure and centrifuge systems which are used to separate oil from olive pastes and avoiding chemical and enzymatic reaction that may changes their sensor properties (Servile *et al*, 2002). Fatty acid represents main physical parameter in chemical structure of olive oil.

Oil which is obtained from pressure equipment represents good quality, does not require water addition to olive paste and rich in flavonids which has an effect on the sensor properties of olive oil. Poor qualities of oil which are lower in free fatty acid compared with good qualities (Giovacchino *et al*, 1994; Kiritsakis, 1998 and Visioli *et al*, 1998).

There are many procedures to determine free fatty acids (FFA) in olive oil, one of these procedures is fourier transforms infrared spectroscopy (FTIR) (Bertran *et al,* 1999). Another advance procedure to detect fatty acid composition and their distribution with high resolution is nuclear magnetic resonance (NMR) which allows the determination of large number of components with a small amount of olive oil samples (Hidalgo *et al*, 2003).

Fatty acid in olive oil components varies according to manufacturing regions and depend on ripeness stage.

Gharsallaoui and his group found that there is no relation between the acidity of olive oil and irrigation system; however it depends on the duration of olive storage (Gharsallaoui *et al*, 2013). Where Martin - Polvillo and his group determined that the storage conditions have an effect on physiochemical properties such as reaction and composite metal particles (Martin-Polvillo *et al*, 1994).

Nierat found that acidity increases with time of storage age, and the relation between acidity and storage age can be represented the relation by fitting the data equation (Nierat *et al*, 2014).

1.3.4 Viscosity of Olive Oil

Viscosity represents one of the most important parameters that are used to deviate the main essential characteristic properties like quality and stability of liquids. Viscosity is represented as a function of temperature and pressure. It measures the resistance to flow or shear (Abramovic *et al*, 1998; liepsch *et al*, 1984 and Viswanath *et al*, 2007).

Viscosity is influenced by many combinations of factors, such as catalyst, temperature, shear rate, molecular weight, pressure and time of storage (Giap *et al*, 2009 and Stanciu, 2012).

Fasina and Hallman studied the viscosity of 12 vegetable oils as a function of temperature, they used three models; modified Williams – Landel Ferry (WLF), power low and Arrhenius. WLF models described the relations between the viscosity of oils and temperature which decreases exponentially and are used to provide oil characteristics (Giap *et al*, 2009 and Fasina *et al*, 2006).

Biresaw and Bantchev studied ten vegetable oils and two petroleum based oils to determine the pressure viscosity coefficient (PVC). They used two different methods: physical properties (viscosity, density) and analysis using Elasto Hydrodynamics (EHD) film thickness, where they noticed that the PVC of vegetable and petroleum – based oils decrease as temperature increases (Biresaw *et al*, 2013).

Abramovic studied dynamic viscosities for numbers of mixture of refined and unrefined vegetable oils (refined sun flower oil, olive oil, refined corn oil and unrefined pumpkin oil), at temperature ranges from 298.15 k up to

328.15 k. He found that the correction constants fitted for dynamic viscosity depend on temperature (Abramovic *et al*, 1998 and Raman, 1923).

Kimilu and his group found that the viscosity of vegetable oil is 15 - 20 times greater than diesel fuel (Kimilu *et al*, 2011).

1.3.5 Refractive Index of Olive Oil

Refractive index represents a fundamental property in physics, chemistry and biology, and is one of the most significant parameter (Yunus *et al*, 2009).

Other researchers compared the refractive index of virgin olive oil and coconut virgin oil. He used digital holographic microscopy with a high degree of accuracy. He found that the refractive index of virgin olive oil is higher than all other oils (Yunus *et al*, 2009).

1.4 Objectives of the Study

The objectives of this study are to:

1) Determine trace elements by using Inductively Coupled Plasma Mass Spectrometry, and compare concentration of metals in different irrigation systems.
2) Study the physical properties of olive oil samples namely; viscosity, acidity, density and refractive index.
3) Compare the physical properties of olive oil samples with standard values.

The samples of olive oil used in this study were collected from different regions in Palestine as follows:

1) Two samples from Hawara town and two other from Misseli village, the samples are named (S_1) whose trees were irrigated from rain. Two samples are from 2013 and two samples are from 2014.
2) A sample (S_2) is watered by natural water which is from Shillo, for the crops of 2014.
3) Three samples from Anabta in Tulkarem, are irrigated by reclaimed waste water for the crops of 2014, these samples are called (S_3).
4) Two samples from Deir Sharaf in Nablus are irrigated by waste water coming from a small valley (Wadi Zomer), and another sample is from Brouqen (Salfit) and it is also irrigated with waste water, and all of these samples with trees irrigated by waste water for the crops of 2014 are named (S_4).

The classification of samples according to their irrigation ways are shown in table (1.3).

Table (1.3): Classification of samples according to their irrigation ways

Region	**Sample**	**Type of Irrigation**	**Crops**
Misseli and Hawara	S_{1a}	Rain water	2013
Misseli and Hawara	S_{1b}	Rain water	2014
Shillo	S_2	Natural water	2014
Anabta	S_3	Reclaimed waste water	2014
Deir Sharaf and Brouqen	S_4	Waste water	2014

Chapter Two

Theoretical Formulation

2.1 Viscosity

Viscosity is one of the main essential physical properties of fluids. It measures the resistance of fluid to shear stress or tensile stress, and it represents the thickness or internal fraction for fluid.

Viscosity depends on structural particle for fluid parameter, correlated with some chemical properties of lipids like the degree of unsaturated fatty acids and their chain length with degrees relation, sensitive to temperature for liquid (Bridgman, 1925; Noureddini *et al*, 1992 and Stanciu, 2012).

2.1.1 Types of Viscosity

There are two types of viscosity which are dynamic and kinematic viscosity.

❖ **Dynamic Viscosity (Absolute Viscosity)**

Dynamic viscosity is defined as resistance to shear and flow, which represents the ratio between shear stress and shear rate.

$$\eta = \frac{\tau}{\gamma} \qquad (2.1)$$

η: it represents dynamic viscosity in Pascal-second (Pa. s), $\gamma = \frac{\partial y}{\partial x}$: is shear rate, known as rate deformation or gradient velocity $(\frac{1}{s})$, $\tau = \frac{F}{A}$: is shear stress, defined as force per unit area $(\frac{N}{m^2}) = \text{Pa}$, equation (2.1) shows that viscosity decreases as shear rate increase (Diamante *et al*, 2014; Dutt *et al*, 2007 and Giap *et al*, 2011).

❖ Kinematic Viscosity

Kinematic viscosity takes place when there is resistance to shear and flow, because of gravity. This type of viscosity defined as ratio of dynamic viscosity to density of fluid which characterized by internal force.

$$\nu = \frac{\eta}{\rho} \tag{2.2}$$

ν is kinematic viscosity in centistokes (cSt) and ρ is mass density of liquid in gm/cm^3.

Equation (2.2) represents mass density of state and deforms the relation between dynamic and kinematic viscosity (Dutt *et al*, 2007). Many features have effects on kinematic viscosity such as bound length between its molecule, number of double bound and its nature (Knothe *et al*, 2005).

2.1.2 Types of Fluids

Fluids including gases, liquids or plasma are substances that flow under shear stress. There are two types of fluids:

a. Newtonian Fluids

The viscosity of Newtonian fluid does not depend on shear force or shear rate. It depends on pressure and temperature. A Newtonian fluid has linear relations with each of shear stress and shear (Chhabra, 2006; Faber, 1995 and Giap *et al*, 2011). Gases, liquids and their solutions that exhibit molecular weight less than 5000 amu and have constant velocity are classified as Newtonian fluids, like water, tea, coffee, honey, edible oils and milk (Chhabra, 2006). Fluids obey equation (2.1) are classified as Newtonian fluids. But it failed to apply all types of fluids (Giap, 2009).

b. Non- Newtonian Fluids

Non`– Newtonian fluids obey either shear stress or shear rate, and do not depend on pressure or temperature. Example of these is helium fluid, it also has difficult formula between shear stress and rate and it has linear relation but does not pass at the origin (Chhabra, 2006 and Faber, 1995).

2.1.3 Units of Viscosity

Viscosity is measured in poise (P) and centipoises (cP), where cP = 0.01P.

Dynamic viscosity is measured in Pascal - second (Pa.s) equivalent in SI units $\frac{N.S}{m^2}$ or $\frac{Kg}{m.s}$ but in cgs unit poise and centipoises named after physicist Jean Louis Marie Poiseuille (1799 - 1869).

kinematic viscosity is measured in SI unit in $\frac{m^2}{s}$, in cgs unit stokes (St), or Centistokes, which is named after George Gabriel Stokes, 1St = 1$\frac{cm^2}{s}$, cSt = 0.01 St (Astle, *et al*, 1988).

2.1.4 Viscosity Theories of Pure Liquids

Viscosity depends on temperature, via Abramovics relation (2.3) (Bridgman, 1925).

$$\eta = \mathrm{A}e^{\frac{B}{T}} \tag{2.3}$$

Equation (2.3) can be written as an Arrhenius relation which defines viscosity as a function of temperature according to equation (2.4) (Diamante *et al*, 2014).

$$\eta = \eta_{\infty,T}\, e^{\frac{E_a}{RT}} \qquad (2.4)$$

η is dynamic viscosity in cP, $\eta_{\infty,T}$ is viscosity at high temperature which is measured in (Pa.s), E_a is activation energy and stands for the stability of liquid deliberates in ($\frac{N.m}{mole}$) or ($\frac{joule}{mole}$), R is the universal gas constant in ($\frac{N.m}{Kelven.mole}$) and T is temperature in Kelvin (Giap *et al*, 2009; Giap *et al*, 2010 and Nierat *et al*, 2014).

Some researchers described the relation between dynamic viscosity and temperature by an additional formula called the William - Landel Ferry formula as represented by:

$$\log \eta = \frac{A}{T} - B \qquad (2.5)$$

where A and B are dimensional constants.

2.2 Refractive Index

Refractive index is a dimensionless quantity, defined as the ratio between the speed of light in vacuum(c) and that in a given medium (v).

$$n = \frac{c}{v} \qquad (2.7)$$

Refractive index is affected by factors such as density, wavelength and temperature of material. Each material has a refractive index that differs from other materials (Thormahlen *et al*, 1985).

2.3 Acidity

Acidity represents the degree of unsaturated fatty acid, and considered as one of the most important parameters that have influence on lipid oxidation (Frega *et al*, 1999). Acidity defines in general formula $CH_3(CH_2)_nCOOH$, it depends on saturated and unsaturated fatty acid according to double bound between molecules.

Single bound is known as saturated fatty acid while double bound is known as unsaturated fatty acid. The acidity represents an important parameter for oil classification. Table (2.1) gives the categorization of olive oil quality according to their acidity (IOOC, 2013 and Qutub *et al*, 2010).

Table (2.1): Categorization of olive oil quality according to % FFA

Category	**% FFA**
Extra Virgin olive oil	≤ 0.8
Virgin olive oil	≤ 2.0
Ordinary virgin olive oil	≤ 3.3
Lampante oil	> 3.3

2.4 Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Inductively Coupled Plasma - Mass Spectrometry (ICP - MS) is an instrument developed in the 1980s. It is used to analyze most of the elements found in the periodic table, trace element and isotopic composition (Date *et al*, 1981; Horn *et al*, 2000; Bosnak *et al*, 2008).

ICP – MS are used especially by industries measurement such as environmental, food, agriculture, semiconductor pharmaceutical, geological, nuclear, forensic chemical, petrochemical and powerful analytical technique with consideration to application of geochemistry (Jenner *et al*, 1990).

2.4.1 Major Component of ICP – MS

ICP – MS device consists of different component which are:

- Sample introduction.
- Ion generation.
- Plasma vacuum interference.
- Ion focusing.
- Ion separation and measurements.

2.4.2 ICP - MS Theory

The sample is introduced into the inductively coupled plasma (ICP) in liquid forms as an aerosol depends on viscosity and volume of samples that are used to analyze the high temperature of plasma, which also dries the aerosol and removes from gas stream using a spray chamber (Houk *et al*, 1980).

A small droplet remains in the argon plasma channel. With a temperature ranging between 6000 – 10000 K, and forms a perfect ion source. High temperature generates high density of positive ions by removing an electron from the components (Houk *et al*, 1980).

Single charged ions with low energy obtained during nebulization are converted to gas as an aerosol and entered into a mass filtering device known as mass spectrometer (Gray, 1975).

ICP - MS system employs quadruple mass spectrometer which is responsible for scanning mass range at rate greater than 5000 atomic mass units (amu) per second.

Ions are separated by their mass - to - charge ratio (m/q) to remain stable and pass through the detector which is responsible for changing the ion density into an electric signal. Other charged ions are ejected and are unstable to passing through the detector. Fig. (2.1) shows how ICP – MS works.

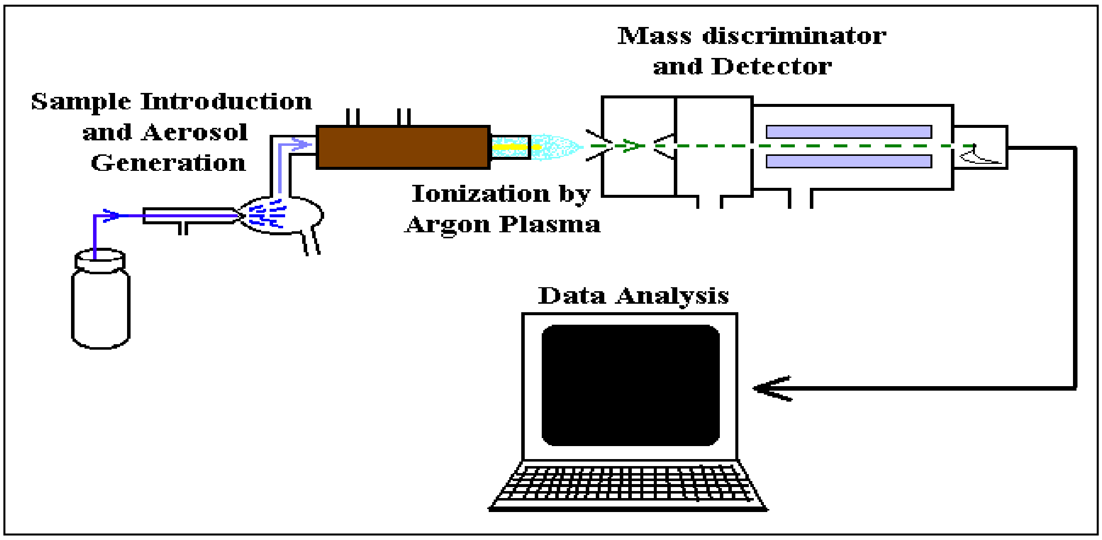


Fig. (2.1): Schematic representation how ICP – MS works Ions are separated and focused by magnetic and electric electrodes, which separate ions according to their charge - to - mass ratio (Batsala *et al*, 2012).

Force is applied on the charged particles in electric and magnetic fields in vacuum to select positive charge ion (q) approximately equal to the charge of electron or one of its multiples. Each ion is subject to Lorentz force Newton's second law :

$$\vec{F} = q(\vec{E} + \vec{V} \times \vec{B}) \qquad (2.8)$$

$$\vec{F} = m\vec{a} \qquad (2.9)$$

Where m is the mass of the ion, a is the acceleration, q is the ion charge, $\vec{E}$ is the electric field, and $\vec{v} \times \vec{B}$ is the vector cross product of the ion velocity and the magnetic field vector.

Positive ions at low voltage accelerate to high speed in terms of kinetic and potential energy according to equation (2.10):

$$\frac{1}{2} m v^2 = q V \qquad (2.10)$$

$$v = \sqrt{\frac{2qV}{m}} \qquad (2.11)$$

When positively charged ions with high speed enter a region of magnetic field perpendicular to their flow, they follow circular path whose radius (R) depends on its mass

$$R = \frac{mv}{qB} = \frac{1}{B}\sqrt{\frac{2Vm}{q}} \qquad (2.12)$$

Where R is the radius of the circular path, B is magnetic field, V is the voltage.

Chapter Three

Methodology

Olive oil samples were collected from different regions in Palestine including Hawara, Misseli, Shillo, Anabta, Deir Sharaf and Brouqen. some samples were from the crops of 2013 and 2014 and the others from 2014 only.

The samples were obtained from trees which were irrigated with different water sources.

The samples for the crops of 2014 were obtained and collected using the same crops collection (traditional way), stored and kept under the same conditions for the crops of 2014 (closed plastic bottles were placed in dark place at room temperature). For each sample three different measurements were taken and the average is represented for measuring the physical properties.

The viscosity of olive samples were obtained and analyzed in wide range of temperature (20.0 – 50.0) $^{\circ}C$. The acidity, density and refractive index were measured in the same range of temperature.

The viscosity of olive oil samples was measured as a function of temperature. The acidity was studied by a titratimetric method.

Trace elements of olive oil samples were measured by using ICP – MS. The tubes of olive oil samples were cleaned with a mixture of distilled water and nitric acid HNO_3 for half an hour. 0.5g from each sample were put in cleaned tubes, then HNO_3were added to dissolve oil and break the links between their molecules. Hydrogen peroxide (H_2O_2) was then added for digestion does not decreases the analytical measurement. Finally samples were heated up to $120.0^{\circ}C$ in stages to reduce deforms of nitrous.

3. Measurement Equipment

3.1 Viscosity Apparatus

Viscosity of olive oil samples was measured using NDJ-1 Rotational Viscometer, which is shown in Fig.(3.1).

Fig.(3.1): NDJ-1 Rotational Viscometer

The viscometer has rotational speeds of 6.0, 12.0, 30.0 and 60.0 RPM, with accuracy of ± 5.0%. Each spindle has an appropriate rotational speed.

The viscosity was measured by determining the spindle zero with suitable rotational speed 60 RPM at different temperature ranges from $20.0^{\circ}C$ to $50.0^{\circ}C$. The spindles measure the viscosity in the range between 1 to 2000000 (cP) with accuracy ∓1%.

3.2 Temperature Apparatus

The temperature of all samples was estimated using Digital prima long thermometer, which is shown in Fig.(3.2). The accuracy of Thermometer is ±1.0%.

Fig.(3.2): Digital Thermometer

3.3 Density Apparatus

The density was measured using Pycnometer shown in fig (3.3). The density was measured by taking the difference mass between a full Pycnometer and an empty one divided by 12.194 cm^3 (volume of olive oil sample), at different temperature with range of $20.0^{\circ}C$ to $50.0^{\circ}C$. The Pycnometer mass was measured using analytical balance with accuracy of $\pm\ 0.00005$.

Fig.(3.3): Pycnometer

3.4 Refractive Index Apparatus

The refractive index was measured using as digital Refractometer at different temperature (20.0 – 50.0)$^{\circ}C$, which is used for measuring different wide range of refractive index at different temperatures. The accuracy of this apparatus is $\pm$0.0002. The digital Refractometer is shown in Fig.(3.4).

Fig.(3.4): Digital Refractometer

3.5 Acidity measurement

The acidity value of olive oil was measured using the titrimetic method. The acidity value represents the mass in miligrams (mg) of potassium hydroxide (KOH) needed to neutralize the free fatty acids in 1gm of olive oil. The samples can be prepered by disolving the oil in a mixture of ethanol – ether, then titrated with standered hydroxide solution, phenol phthalien is also used as an indicator.

The acidity of olive oil samples are measured and three main steps are used to prepare the mixture which they are:

1) 0.1 M of KOH mixture is prepared according to these steps:

- 0.56 g of solid KOH transferred into volumtric flask to be crushed and converted to powder. Then this amount of powder is disolved in100 mL of absolute ethanol to get 0.01 moles with 0.1M of KOH ethanol solution. The molarity of KOH can be found according to the equation (3.1):

$$\text{Molarity} = \frac{weight\ of\ KHp}{molecular\ weight\ of\ KHp} \times \frac{1000}{volume\ of\ KOH\ (ml)} \qquad (3.1)$$

Additionally, the number of moles can be found by applying the relation (3.2):

$$\text{Number of moles} = \frac{\text{weight}}{\text{molecular weight}} \qquad (3.2)$$

- 0.204 g of solid KHP is weighted and transferred into a conical flask to be disolved completely in 50.0 mL of distilled water. This step is used to get 0.01 mole with 0.2 M of KHP.
- 3 drops of phenophathalein are added for titrated KOH.
- Mixture of KHP are added to KOH with few droplet of phenol phthelen approximatly 3 droplet untill the colour of mixture is changed to pink and persisted for 30 second.
- Step three is repeated 3 times for titrite KHP.

2) Ethanol – ether mixture was prepared:
   - 50.0 mL of ethanol is mixed with 50.0 mL ether and 3 drops of phenol phthelen were added to the mixture.
   - A Few drops of ethanolic KOH are added to get the pink color.
3) Acid value of olive oil is obtained:
   - 5-10 g of olive oil samples are weighted in a conical flask.

- Ethanol – ether mixture with a concentration of 1:1 are added.
- 3 drops of phenol phthalen are added.
- The resulting solution titrated with standard KOH with a permanent faint pink color that appears for a few seconds for olive oil samples (Nierat, 2012).

Finally the acid values of olive oil samples and free fatty acid (FFA) are determined according to equations (3.3 and 3.4):

$$\text{Acid value} = \frac{standerd\ solution\ KOH(ml) \times molarity\ KOH \times 56.1}{weight\ of\ sample(g)} \tag{3.3}$$

$$\%\ \text{FFA} = \frac{\text{Acid value}}{1.99} \tag{3.4}$$

3.6 Trace Elements Apparatus

The trace element of olive oil samples were determined by using ICP – MS (Perkin Elmer Elan 9000) method, which represents an inorganic device and changes the atoms of elements in olive oil samples to ion distinguished by using mass spectrometer (Baysal *et al*, 2013). This device is used to detect and analyze the nature of samples according to trace elements which are found in olive oil samples. Figure (3.5) shows ICP – MS device.

Figure (3.5): ICP – MS (Perkin Elmer Elan 9000) device

ICP - MS performs at high sensitivity, good control of interferences, analysis with excellent sensitivity and higher throughput than other devices (Hieftie *et al*, 1992). ICP - MS are used for trace elements to determinate from part per trillion (PPt) to part per million (PPm).

Chapter Four

Results and Data Analysis

4.1 The Physical Properties of Samples with Trees Depend on Rain Water of Crop 2014

4.1.1 Mass Density Results

The mass density results in (gm/cm^3) of olive oil sample (S_{1b}) from Misseli village and Hawara town, of crop 2014 at different temperatures, three different measurements were taken and the average is shown in table (4.1)

Table (4.1): Measured mass density in (gm/cm^3) of olive oil sample (S_{1b}), with trees depend on rain water of crop 2014

T(°C)	Density in (gm/cm^3)
20.0	0.9169
27.2	0.9124
30.0	0.9100
35.0	0.9064
36.8	0.9050
39.7	0.9021
43.2	0.8992
45.0	0.8977
50.0	0.8942

The relation between the densities of olive oil and temperatures of sample (S_{1b}), in which they depend on rain water of crop 2014 are given in Fig.(4.1).

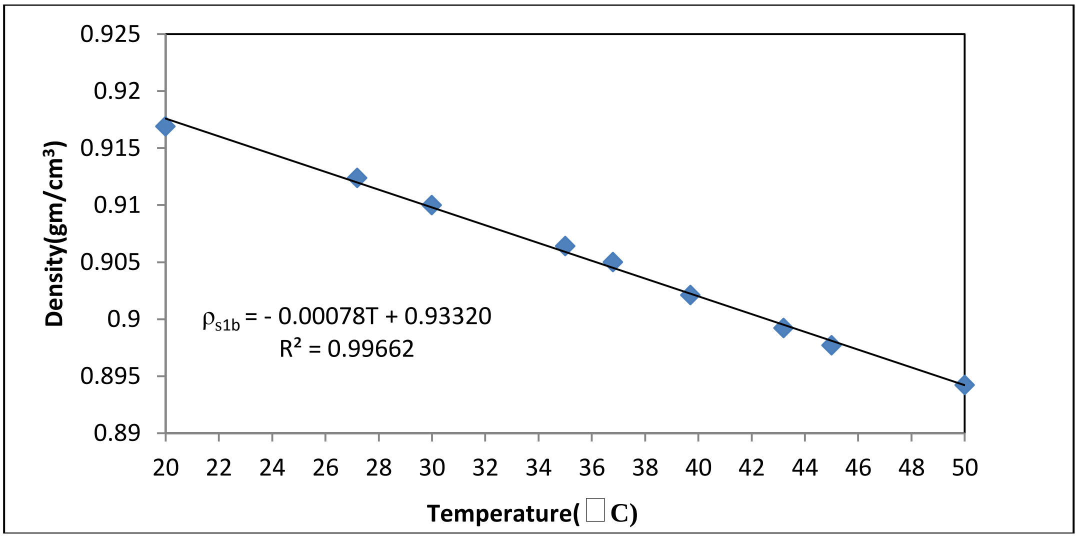


Fig.(4.1): The measured mass density of olive oil samples (S_{1b}), with trees depend on rain water at different temperatures of crop 2014

Fig.(4.1) shows that the slope for density of sample (S_{1b}) is 0.00078 gm/°C.cm^3, which depends on rain water. The measured density decreases as a function of temperature. At 20.0°C our measured data is 0.9169 gm/cm^3, while the range of standard value is (0.910 - 0.916) gm/cm^3 (Codex Standard, 2001 and Palestinian Standard, 1997).

4.1.2 Viscosity Results

The viscosity of the olive oil samples (S_{1b}) with trees depends on rain water at different temperatures from 20.0°C up to 50.0□C of crop 2014 are given in table (4.2).

Table (4.2): Measured viscosity of olive oil samples (S_{1b}), with trees depend on rain water of crop 2014

T(°C)	Viscosity in (cP)
20.0	69.5
22.0	62.1
24. 0	55.9
26.0	50.6
28.0	45.3
30.0	41.0
32.0	36.8
34.0	33.4
36.0	30.0
38.0	27.0
40.0	24.4
42.0	22.3
44.0	20.1
46.0	17.8
48.0	15.8
50.0	15.4

The viscosity of olive oil sample of crop 2014 from (S_{1b}) as a function of temperature is plotted and shown in Fig.(4.2).

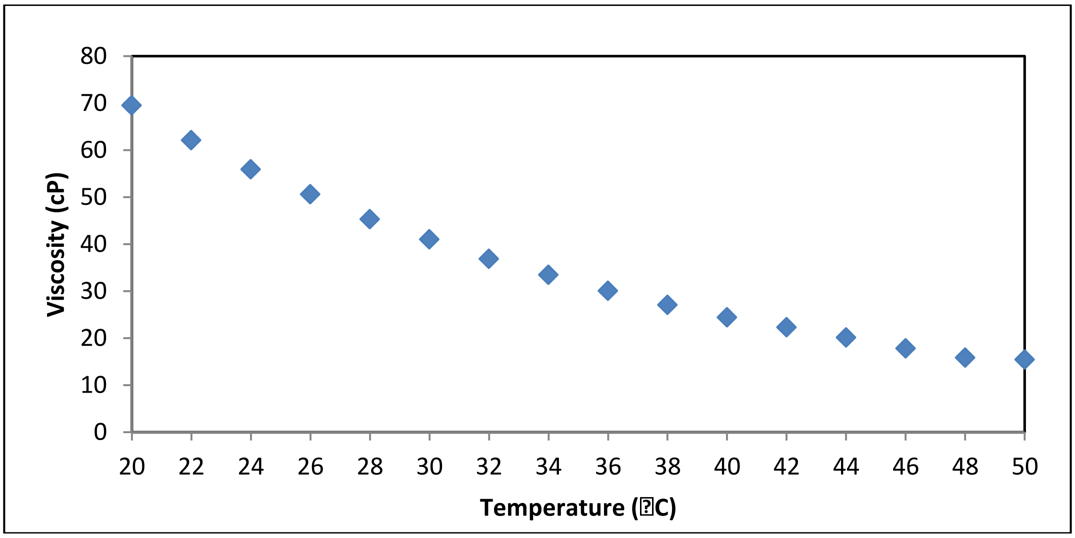


Fig.(4.2): The viscosity measured of sample (S_{1b}), with trees depend on rain water at different temperature of crop 2014

The viscosity decreases inversely proportion as a function of temperature as shown in Fig.(4.2). Our measured viscosity at 20. 0 °C is 69.5 cP, which is considered to be the standard of our different measurements.

4.1.3 Refractive Index Results

The refractive index of olive oil samples from two different locations Hawara town and Misseli village (S_{1b}) of crop 2014 were measured as a function of temperature. The measured data are given in table (4.3).

Table(4.3): Measured refractive index of olive oil sample (S_{1b}), with trees depend on rain water of crop 2014

T(°C)	Refractive Index
20.5	1.4670
25.8	1.4662
32.2	1.4655
37.0	1.4649
41.0	1.4645
47.4	1.4638
50.0	1.4636

The refractive indices of olive oil sample (S_{1b}) as a function of temperature are plotted in Fig.(4.3).

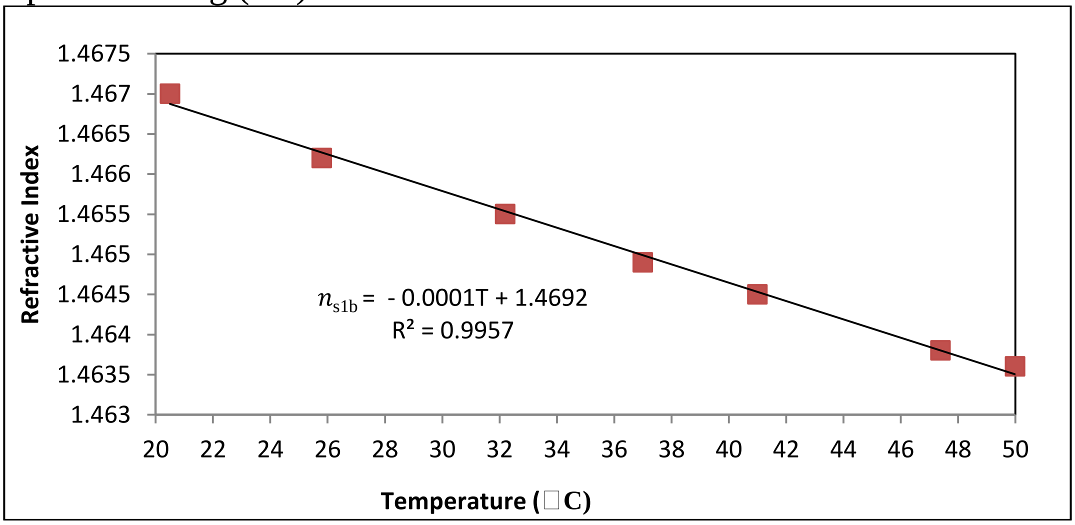


Fig.(4.3): Measured refractive index of olive oil sample (S_{1b}), with trees depend on rain water at different temperatures of crop 2014

The refractive index is affected by temperature with slope -0.0001/ ℃ for (S_{1b}) sample. At 20℃ our measured refractive index is 1.4670, which is in the range of standard value (1.4677 - 1.4706) (Codex Standard, 2001).

4.2 The Physical Properties of Samples with Trees Irrigated by Natural Water of Crop of 2014

4.2.1 Mass Density Results

The mass density results at different temperatures of olive sample (S_2) from Shillo, with trees irrigated by pure water are given in table (4.4).

Table(4.4): Measured mass density in (gm/cm^3) of olive oil samples (S_2), with tress irrigated by natural water of crop 2014

T(℃)	Density in (gm/cm^3)
20.2	0.9179
25.2	0.9143
30.9	0.9092
33.2	0.9073
35.3	0.9059
37.2	0.9041
39.5	0.9020
44.1	0.8995
49.8	0.8941

The measured data for densities of olive oil sample (S_2) are fitted as a function of temperature in Fig. (4.4).

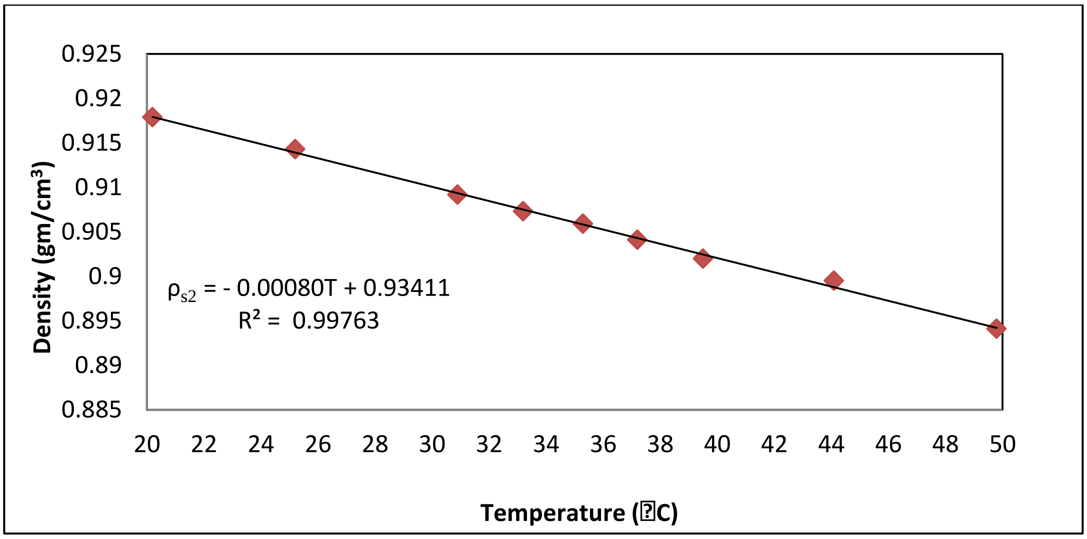


Fig.(4.4): The density of olive oil sample (S_2), with trees were irrigated by natural water at different temperature of crop 2014

Fig.(4.4) gives the slope for density of sample S_2 to be -0.00080 gm/°C.cm^3. The measured density decreases as a function of temperature. At 20°C our measured density is 0.9179 gm/cm^3, and this measured value is near the standard value (0.910 – 0.916) gm/cm^3 (Codex Standard, 2001 and Palestinian Standard, 1997).

4.2.2 Viscosity Results

The viscosity of olive oil of crop 2014 from S_2, with trees irrigated by natural water as a function of temperature was measured. The measured values of viscosity are tabulated in table (4.5).

Table(4.5): The measured viscosity of olive oil sample (S_2), with trees irrigated by natural water of crop 2014

T(℃)	Viscosity in (cP)
20.0	72.3
22.0	64.9
24.0	59.2
26.0	53.2
28.0	48.4
30.0	43.9
32.0	40.0
34.0	36.4
36.0	33.2
38.0	30.3
40.0	27.9
42.0	25.4
44.0	23.4
46.0	21.3
48.0	19.5
50.0	18.7

The viscosity of olive oil of crop 2014 from sample (S_2) at different temperatures is shown in Fig.(4.5).

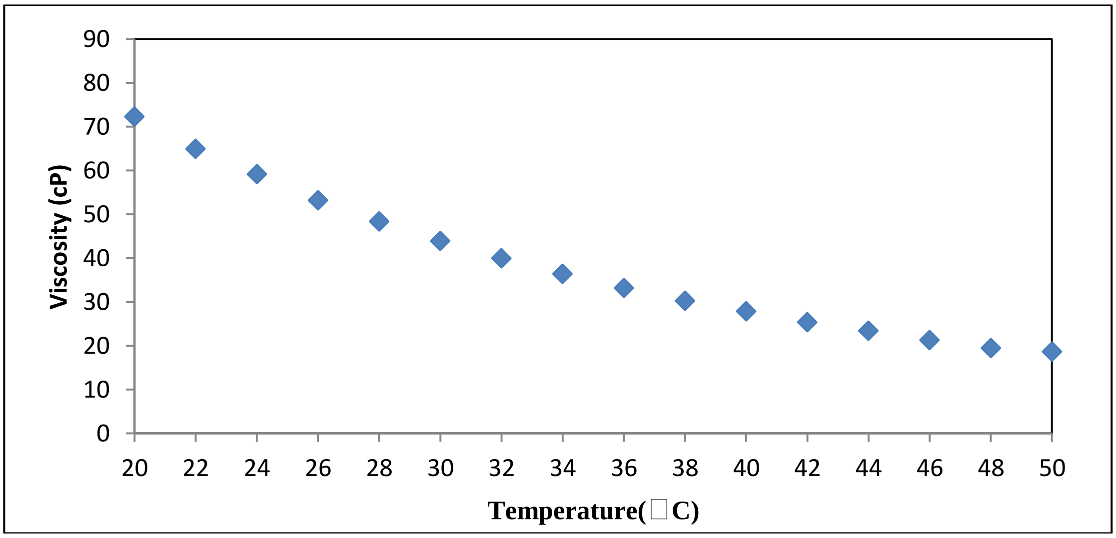


Fig.(4.5): The relation between viscosity of olive oil sample (S_2), with trees irrigated by natural water and temperatures of crop 2014

The inverse relationship between viscosity and temperatures is shown in Fig.(4.5). At $20.0°C$ the measured viscosity of sample (S_2) is 72.3 cP, which is greater than 69.5 cP for standard value of samples depend on rain water.

4.2.3 Refractive Index Results

The measured refractive index values of sample (S_2), with trees irrigated by natural water of crop 2014 are given in table (4.6).

Table(4.6): Measured refractive index of sample (S_2), with trees irrigated by natural water for the crops of 2014

T(°C)	Refractive Index
20.0	1.4675
23.3	1.4671
26.3	1.4669
32.4	1.4664
42.1	1.4655
49.5	1.4649
51.4	1.4647

The relation between refractive index versus temperature of crop 2014 is shown in Fig.(4.6).

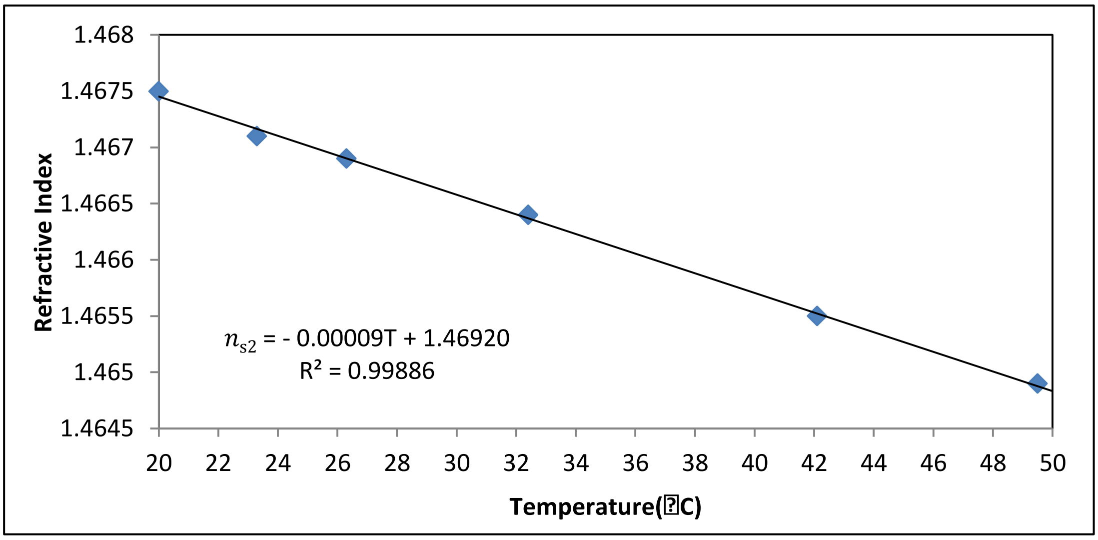


Fig.(4.6): The measured refractive index of sample (S_2), with trees irrigated by natural water at different temperatures of crop 2014

Fig.(4.6) gives the slope of refractive indices for S_2 to be -0.0009/ °C, the measured refractive index for irrigated samples by natural water at 20. 0°C is 1.4675, it is in the range of standard value (1.4677 – 1.4706) and greater than samples depend on rain water (Codex Standard, 2001).

4.3 The Physical Properties of Samples with Trees Irrigated by Reclaimed Waste Water of Crop 2014.

4.3.1 Mass Density Results

The mass density result in (gm/cm^3) of olive oil sample (S_3) from different three location from Anabta in Tulkarem, with trees irrigated by reclaimed waste water of crop 2014 at different temperatures. The average values are given in table (4.7).

Table(4.7): Measured mass density in (gm/cm^3) of olive oil samples (S_3), with trees irrigated by reclaimed waste water at different temperatures of crop 2014

T(°C)	Density in (gm/cm^3)
20.3	0.9198
21.9	0.9182
27.6	0.9142
29.4	0.9121
31.5	0.9101
36.7	0.9064
40.0	0.9036
44.4	0.9003
49.0	0.8968

The relation between the densities of olive oil samples and temperature of sample (S_3), with trees irrigated by reclaimed waste water of crop 2014 is shown in Fig.(4.7).

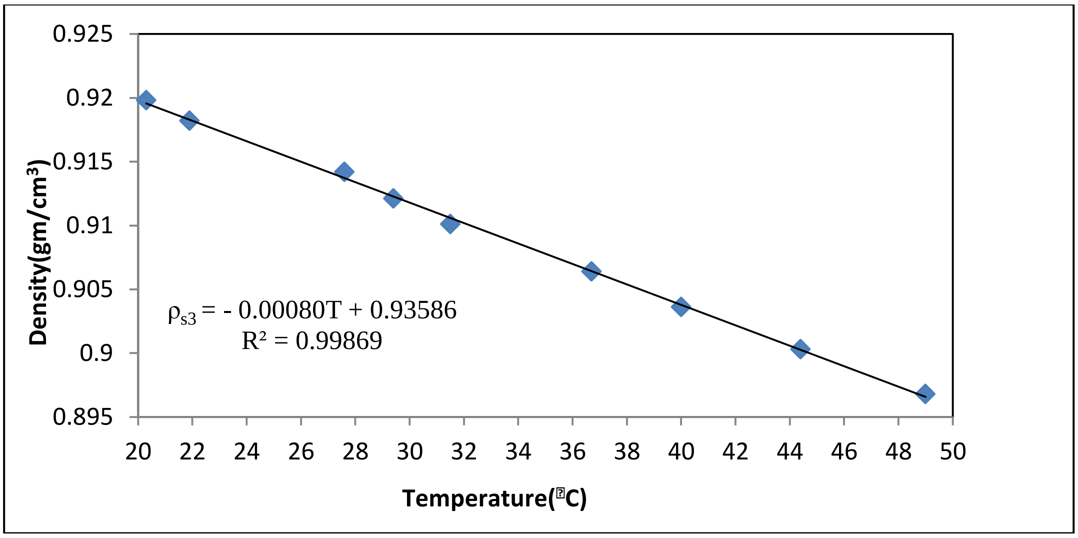


Fig.(4.7): The measured density of sample (S_3), with trees irrigated by reclaimed waste water at different temperatures of crop 2014

The density of sample (S_3) with trees irrigated by reclaimed waste water decreases linearly as a function of temperature and their slope is -0.00080 gm/°C.cm^3 as shown in Fig.(4.7). The error of measuring density is ∓ 0.0005 gm/cm^3. The measured density at 20.0°C is 0.9198 gm/cm^3, this measured value is greater than standard value (0.910 – 0.916) gm/cm^3 (Codex Standard, 2001 and Palestinian Standard, 1997) and is greater than samples depend on rain water and is irrigated by natural water.

4.3.2 Viscosity Results

The viscosity result for samples (S_3), with trees irrigated by reclaimed waste water, measured at different temperature measured in cP of crop 2014 are given in table (4.8).

Table(4.8): Measured viscosity of olive oil samples (S_3), with trees irrigated by reclaimed waste water at different temperatures of crop 2014

T(°C)	Viscosity in (cP)
20.0	73.2
22.0	66.2
24.0	59.6
26.0	53.2
28.0	47.9
30.0	43.6
32.0	39.4
34.0	35.6
36.0	31.7
38.0	28.6
40.0	25.7
42.0	23.5
44.0	21.0
46.0	19.1
48.0	17.2
50.0	16.3

The measured values of viscosity of olive oil sample (S_3) of crop 2014 are plotted in Fig.(4.8).

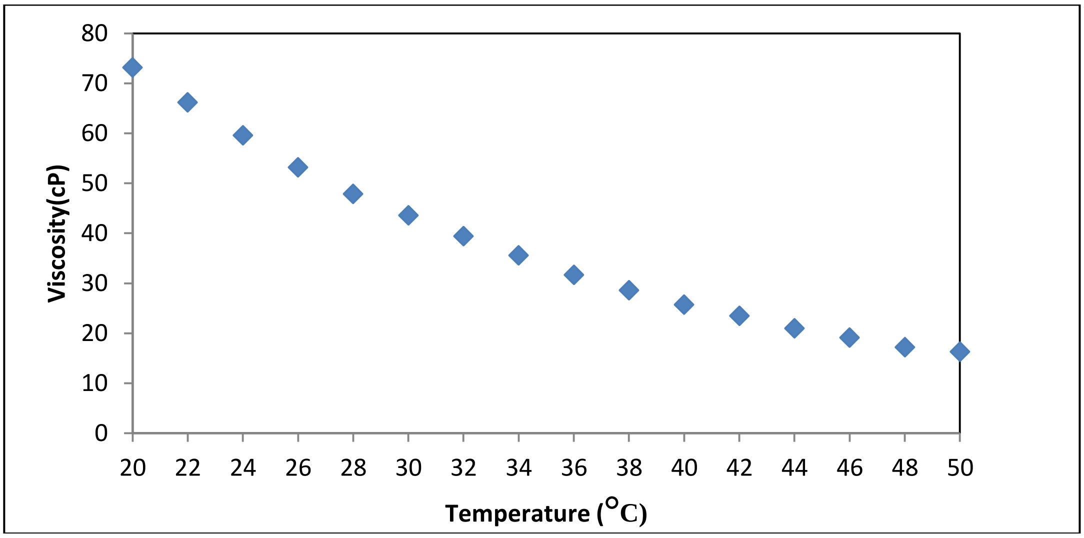


Fig.(4.8): The viscosity measured of sample (S_3), with trees irrigated by reclaimed waste water at different temperatures of crop 2014

Fig.(4.8) shows the viscosity decreases when the temperature increases. The measured viscosity at 20.0℃ is 73.2 cP, which is greater than the viscosity of samples depend on rain water (69.5 cP).

4.3.3 Refractive Index Results

The measured refractive indices of olive oil samples (S_3), at different temperatures of crop 2014 are given in table (4.9).

Table (4.9): Measured refractive index of olive oil sample (S_3), with trees irrigated by reclaimed waste water at different temperatures of crop 2014

T(°C)	Refractive Index
20.0	1.4672
24.3	1.4669
26.5	1.4667
30.6	1.4662
35.0	1.4659
37.5	1.4656
43.0	1.4650
50.0	1.4645

The relationship between refractive indices of olive oil sample (S_3) at different temperatures is plotted in Fig. (4.9).

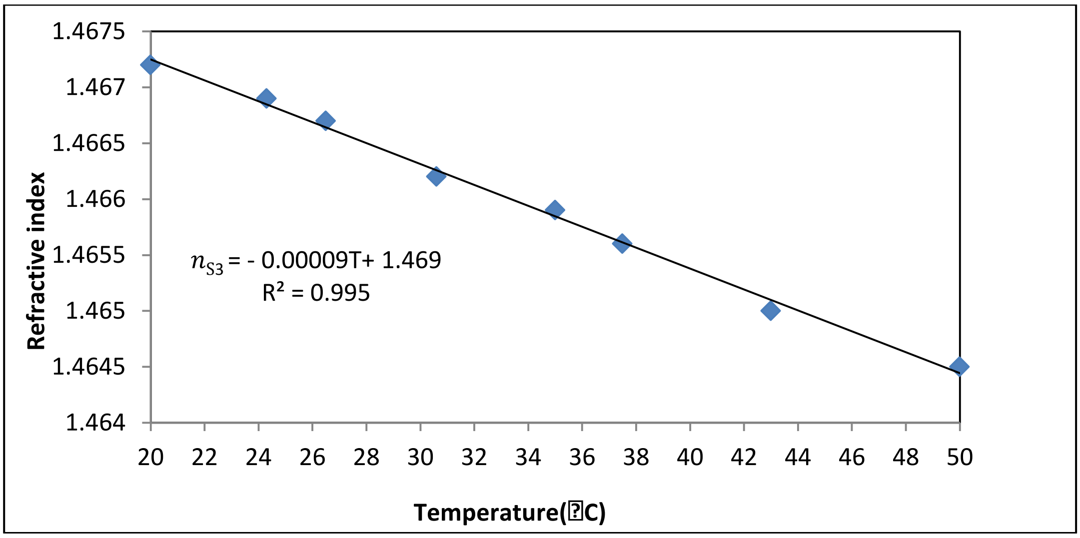


Fig.(4.9): Measured refractive index of sample (S_3), with trees irrigated by reclaimed waste water at different temperatures of crop 2014

In Fig.(4.9) the refractive index decease with slope -0.00009/ °C. Our measured refractive index at 20°C is 1.4672 in the range of standard values (1.4677 – 1.4706) (Codex Standard, 2001).

4.4 The Physical Properties of Samples which their Trees Irrigated by Waste Water of Crop 2014

4.4.1 Mass Density Results

The measured mass density of olive oil sample (S_4), from Deir Sharaf in Nablus and from Brouqen in Salfit, which their trees irrigated by waste water, at different temperatures of crop 2014 are given in table (4.10).

Table (4.10): Measured mass density in (gm/cm^3) of olive oil sample (S_4), with tress irrigated by waste water at different temperature of crop 2014

T(°C)	Density in (gm/cm^3)
20.6	0.9205
23.6	0.9183
28.1	0.9136
30.7	0.9108
33.9	0.9086
40.0	0.9042
43.6	0.9012
46.0	0.8986
49.5	0.8960

The density of olive oil sample (S_4) with trees irrigated by waste water of crop 2014 is shown in Fig. (4.10).

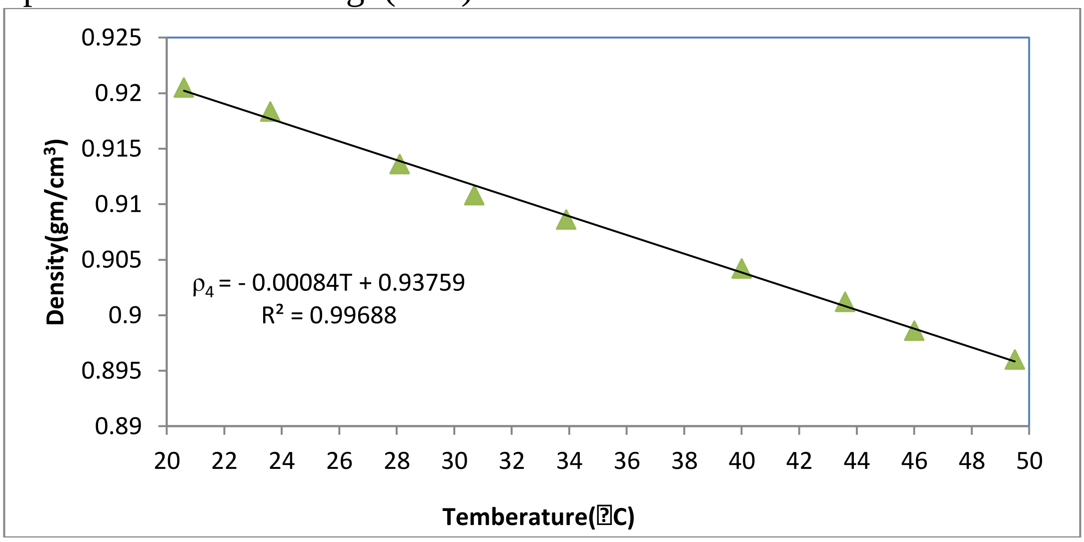


Fig.(4.10): The density of sample (S_4), with trees irrigated by wastewater at different temperature of crop 2014

The relationship between the densities of olive oil sample (S_4) and change in temperatures are linear, and also the density decreases as a function of temperature as shown in Fig.(4.10). the slope for sample (S_4) is -0.00084 gm/°C.cm^3. At 20°C our measured value is 0.9205gm/cm^3, which is greater than standard value (0.910 – 0.916) (Codex Standard, 2001). The measured values appears that the density increases for irrigated samples by waste water.

4.4.2 Viscosity Results

The measured viscosity of sample (S_4), with trees irrigated by waste water of crop 2014 is given in table (4.11).

Table (4.11): Measured viscosity of olive oil sample (S_4), with trees irrigated by waste water of crop 2014

T(°C)	Viscosity in (cP)
20.0	80.4
22.0	73.6
24.0	67.3
26.0	61.8
28.0	57.1
30.0	51.9
32.0	47.9
34.0	43.8
36.0	40.7
38.0	38.0
40.0	35.2
42.0	33.0
44.0	30.8
46.0	28.9
48.0	26.8
50.0	25.2

The relation between viscosity of olive oil sample (S_4) and temperature of crop 2014 is shown in Fig.(4.11).

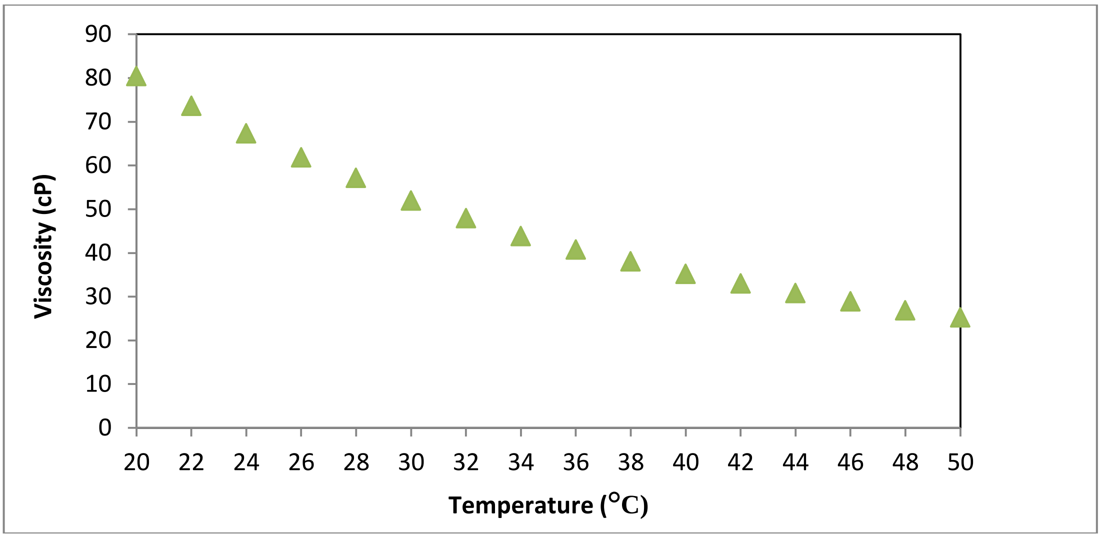


Fig.(4.11): The viscosity of sample (S_4), with trees irrigated by waste water at different temperatures of crop 2014

Fig.(4.11) describes the temperature dependence of viscosity. At 20.0℃ our measured viscosity is 80.4 cP which is greater than value of sample depends on rain water(69.5 cP at 20.0 ℃). The trend of viscosity of samples increases for samples were irrigated by waste water.

4.4.3 Refractive Index Results

The measured value of the refractive index of sample (S_4), with trees were irrigated by waste water of crop 2014 at different temperatures are given in table (4.12).

Table (4.12): Measured refractive index of olive oil sample (S_4), with trees irrigated by waste water at different temperatures of crop 2014

T(°C)	Refractive Index
20.0	1.4674
23.3	1.4671
29.8	1.4664
34.5	1.4660
41.2	1.4654
45.8	1.4651
50.0	1.4648

The relations between refractive indices of olive oil samples (S_4) at different temperatures are given in Fig.(4.12).

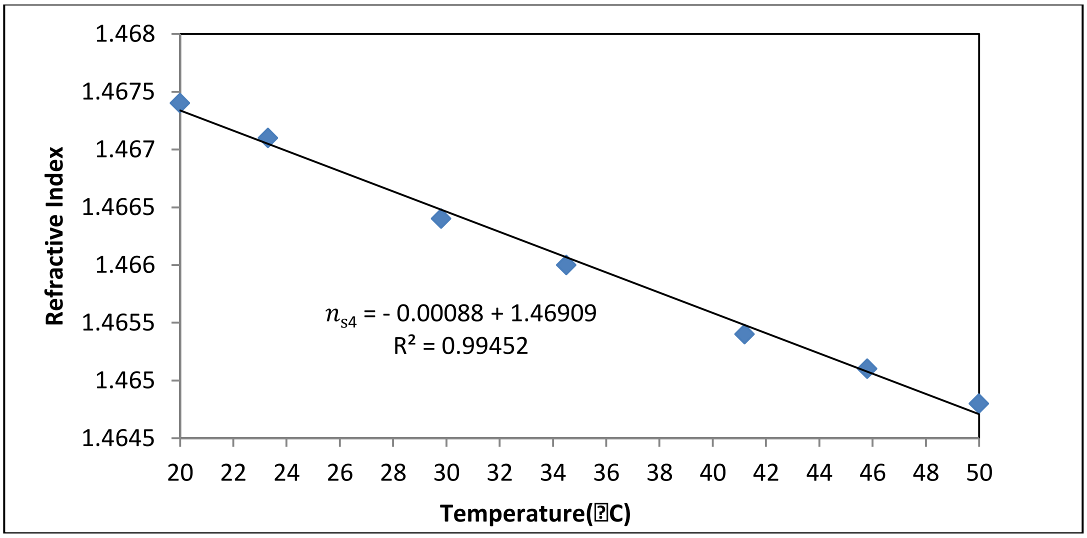


Fig.(4.12): Measured refractive index of olive oil sample (S_4), with trees irrigated by waste water at different temperatures of crop 2014

The refractive index decreases as a function of temperature with slope -0.00088/ °C as shown in Fig. (4.12). The measured value at 20.0°C is 1.4674 for irrigated samples by waste water. This measured value is in the range of standard values (1.4677 – 1.4706) (Codex Standard, 2001). The refractive index increases for irrigated sample by waste water.

4.5 Comparing between the Physical Properties of Different Samples According to their Irrigation System

4.5.1 Mass Density Results

The mass densities of different olive oil samples were measured at 20. 0℃ of crops 2013 and 2014, and compared with codex standard are given in table (4.13) (Codex Standard, 2001). Density as a function of temperature of different samples are shown in Appendix A.

Table(4.13): Our measured mass density in (gm/cm^3) of olive oil for different samples at 20. 0 ℃ compared with codex standard

Region	Sample	Type of Irrigation	Crops	Our Density	Codex Standard
Hawara and Misseli	S_{1a}	Rain water	2013	0.9161	0.910 - 0.916
Hawara and Misseli	S_{1b}	Rain water	2014	0.9169	
Shillo	S_2	Natural water	2014	0.9179	
Anabta	S_3	Reclaimed waste water	2014	0.9198	
Deir Sharaf and Brouqen	S_4	Waste water	2014	0.9205	

The average density in gm/cm^3 of olive oil samples (S_{1b}) for the crops of 2014 is 0.9169 gm/cm^3 compared with crops of 2013 which was 0.9161 gm/cm^3. The highest value of measured density is 0.9205 gm/cm^3 for sample S_4, which their trees were irrigated by wastewater of crop 2014 and it is greater than standard value (0.910 – 0.916) gm/cm^3. The lowest values of measured densities were 0.9161 gm/cm^3 for sample S_{1a} which their trees depend on rain water, and it is in good agreement with standard value (0.910 – 0.916) gm/cm^3. The density increases for irrigated samples by waste water compared with samples irrigated by rain water.

The relationship between densities of olive oil samples and temperature are given in Fig.(4.13) for different irrigation system.

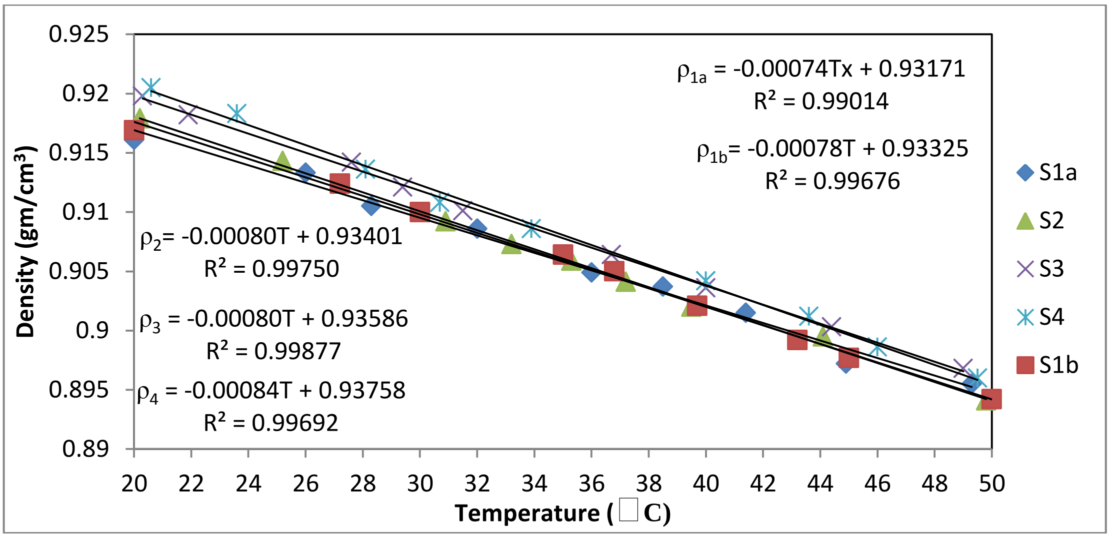


Fig.(4.13): The measured density of olive oil samples for different irrigation system

4.5.2 Viscosity Results

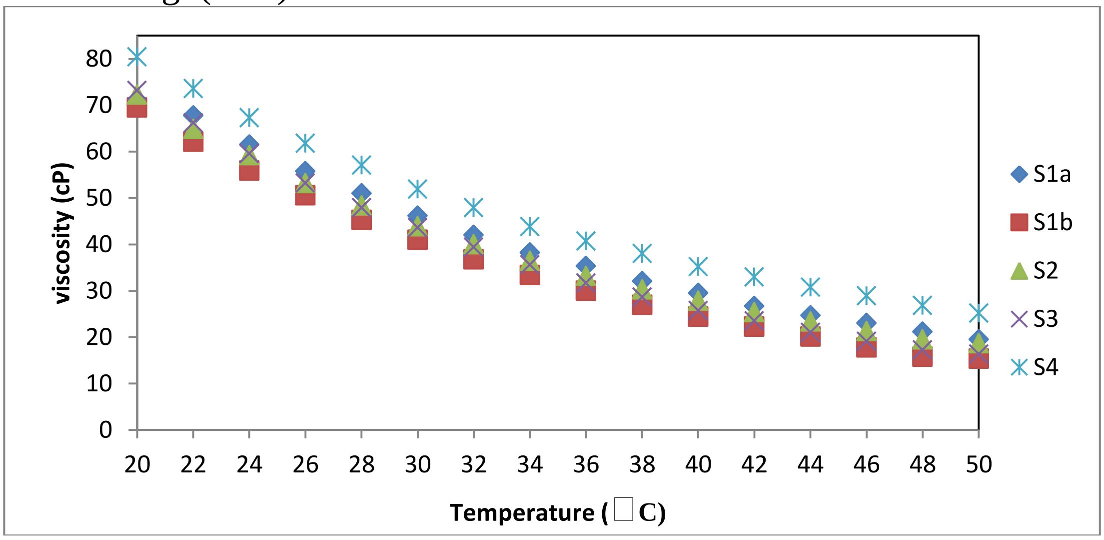
The viscosity of olive oil samples decreases with increasing temperature, this is because when the temperature increase it causes to decrease the inter molecular forces between the nearest neighbors molecule of samples. The measured results for the viscosity of olive oil samples were measured at 20.0℃ of crop 2014 are given in table (4.14).

Table(4.14): The measured viscosity in cP of olive oil for different samples at 20. 0℃

Region	Sample	Type of Irrigation	Crops	Viscosity
Hawara and Misseli	S_{1a}	Rain water	2013	70.2
Hawara and Misseli	S_{1b}	Rain water	2014	69.5
Shillo	S_2	Natural water	2014	72.3
Anabta	S_3	Reclaimed waste water	2014	73.2
Deir Sharaf and Brouqn	S_4	Waste water	2014	80.4

The viscosity of samples with trees irrigated by waste water are greater than viscosity of other irrigation system. Viscosity as a function of temperature of different samples are shown in Appendix B.

The viscosity for different samples according to their irrigation system are shown in Fig. (4.14)



Fig(4.14): The measured viscosity of olive oil samples for different irrigation system

4.5.3 Refractive Index Results

The measured refractive index for all olive oil samples were measured at room temperature 23. 0°C, of two different crops 2013 and 2014 and compared with standard value are given in table (4.15) (International Olive Council, 2011; Codex Alimentarius, 2001).

Table(4.15): Our measured refractive index of olive oil of different samples at 23.0℃ compared with standard value

Region	Sample	Type of Irrigation	Crops	Refractive Index	Codex Standard
Misseli and Hawara	S_{1a}	Rain water	2013	1.4656	1.4677 - 1.4706
Misseli and Hawara	S_{1b}	Rain water	2014	1.4665	
Shillo	S_2	Natural water	2014	1.4671	
Anabta	S_3	Reclaimed waste water	2014	1.4670	
Deir Sharaf and Brouqen	S_4	Waste water	2014	1.4671	

The measured refractive index of samples which irrigated by natural water, reclaimed waste water and waste water are greater than samples that depends on rain water.

4.5.4 Acidity Results

The acidity results of olive oil samples of two different crops 2013 and 2014 from different location was measured.

The measured values are given in table (4.16). The classification of olive oil samples is given according to table (2.1).

Table (4.16): The acidity results and classification of olive oil samples of two different crops 2013 and 2014

Region	Type of Irrigation	Sample	Crops	%FFA	Olive Oil Classification
Misseli and Hawara	Rain water	S_{1a}	2013	2.34	Ordinary virgin olive oil
Misseli and Hawara	Rain water	S_{1b}	2014	1.85	Virgin olive oil
Shillo	Natural water	S_2	2014	2.73	Ordinary virgin olive oil
Anabta	Reclaimed waste water	S_3	2014	3.03	Ordinary virgin olive oil
Deir Sharaf and Brouqen	Waste water	S_4	2014	3.35	Lampante oil

Table (4.16) shows most of olive oil samples from different location are differ in their acidity. Samples S_{1b} for crops of 2014 are virgin olive oil, while samples (S_2 and S_3) of crop 2014 and sample (S_{1a}) of crop 2013 form ordinary virgin olive oil while S_4 are Lampante oil. The acidity of olive oil affected by different factors such as method and period of storage oil, cultural techniques employed for oil extraction and irrigation system.

The measured values of acidity of olive oil samples from different location are plotted in Fig.(4.15).

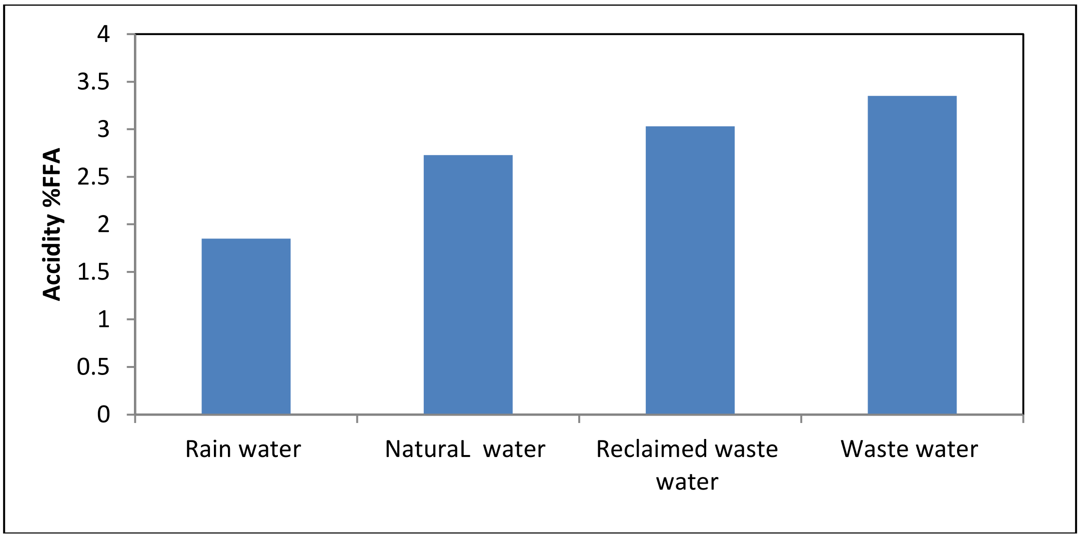


Fig.(4.15): The relation between acidity and type of samples according to their irrigation system of crop 2014

Fig.(4.15) shows that the acidity for samples with tress irrigated by waste water are greater than other samples.

The results for acidity of olive oil samples determine the quality of olive oil which varys between virgin for samples depend on rain water, ordinary virgin for irrigated samples by reclaimed waste water and Lampante oil for irrigated samples by waste water. The classification of olive oils are due to their acidity according to table (2.1).

4.6 Trace Elements

The results for metals concentration of olive oil samples for different sites according to their irrigation system of crops 2013 and 2014 are given in table (4.17).

Table (4.17): Results for metals concentration (µg/g) in olive oil samples, which they were measured by using ICP – MS for different irrigation system

Metal	Rain Water of 2013	Rain Water of 2014	Natural Water of 2014	Reclaimed Waste Water of 2014	Waste Water of 2014
Ca	1164.825	1110.6730	949.0872	1317.1110	1836.8490
Cd	0.0778	0.0898	0.0036	0.0108	0.0110
Co	0.0408	0.0198	0.0124	0.0140	0.0202
Cr	2.6076	2.1432	2.1632	2.1028	2.1116
Cu	1.4352	1.1830	1.1058	1.4316	1.7400
Fe	33.8176	26.9542	22.2520	25.7286	43.4490
K	25.9488	22.7490	15.4688	21.2264	38.6888
Mg	257.2330	301.3100	270.6774	519.0152	637.9324
Mn	0.7702	0.4830	0.2644	0.4324	0.6836
Na	234.8748	171.4816	115.8286	225.2206	247.9686
Ni	0.7512	0.2754	0.0892	0.1784	0.2890
Pb	1.6348	3.5768	0.5182	0.8388	0.6818
Zn	13.4984	8.0478	7.4032	11.3044	23.7126

The determination of trace metals in olive oil samples by using ICP – MS can be used for the characterization of oil quality by estimating the concentration of metals, which shows difference for each oil concentration according to their irrigation system.

4.6.1 Calcium (Ca)

Calcium is a useful metal for building bones and teeth for human body, a lack of calcium can cause osteoporosis dieses (Christiansen *et al*, 1975).

The concentrations of Ca according to their irrigation system are shown in Fig. (4.16).

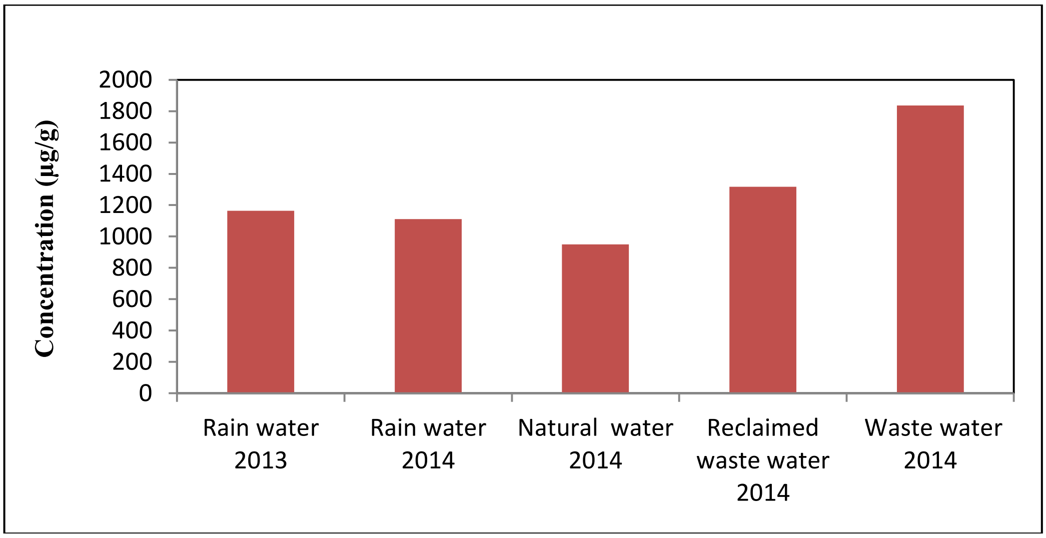


Fig.(4.16): Concentration of Calcium in olive oils for all sampling sites according to their irrigation system of crops of 2013 and 2014

The Fig.(4.16) shows that the (Ca) content of the samples ranged from 949.0872 to 1836.849 µg/g, Shillo site which their trees irrigated by natural water had the lowest concentration whereas Brouqen with trees irrigated by waste water had the highest concentration. The concentrations of calcium in this study is greater than previous studies according to table (1.1).

4.6.2 Sodium (Na)

Sodium is an essential trace element and its required for body in a determine concentration, higher concentration of Na that causes high blood pressure (Washington, 2010). The concentrations of Sodium according to their irrigation system, which they are shown in Fig. (4.17).

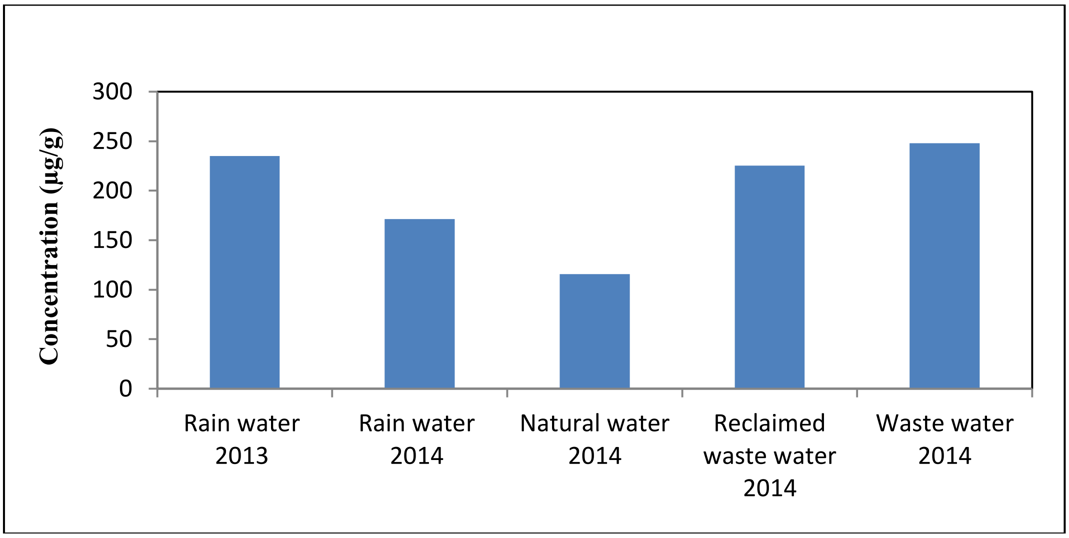


Fig.(4.17): Concentration of Sodium in olive oils for all sampling sites according to their irrigation system of crops of 2013 and 2014

The highest concentration of Na for sample S_4 with trees irrigated by waste water is 247.9686 µg/g, but the lowest value of sample S_2, their trees irrigated by natural water as shown in Fig.(4.17). The concentration of sodium in all our samples is higher comparing with previous studies according to table (1.1).

4.6.3 Potassium (K)

Bodies need potassium to build muscles and control the electrical activity of the heart, higher concentration can cause abnormal heart disease (Washington, 2010). The concentrations of potassium according to their irrigation system, which they are shown in Fig. (4.18).

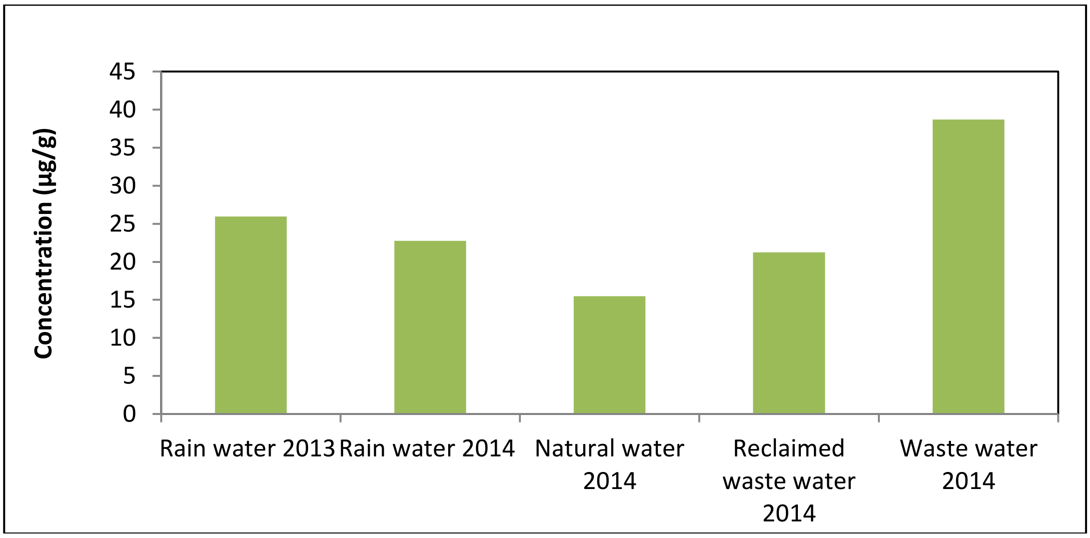


Fig.(4.18): Concentration of Potassium in olive oils for all sampling sites according to their irrigation system of crops of 2013 and 2014.

The maximum value of K is 38.6888 µg/g of sample S_4 with trees irrigated by waste water, while the minimum value is 15.4688 µg/g of sample S_2 from Shillo site with trees irrigated by natural water.

The results for the concentration of potassium metal are greater than previous studies which are illustrated in table (1.1).

4.6.4 Magnesium (Mg)

Magnesium is one of essential elements for human body. The body needs Mg at high concentration compared to other metals. The concentrations of magnesium according to their irrigation system are shown in Fig. (4.19).

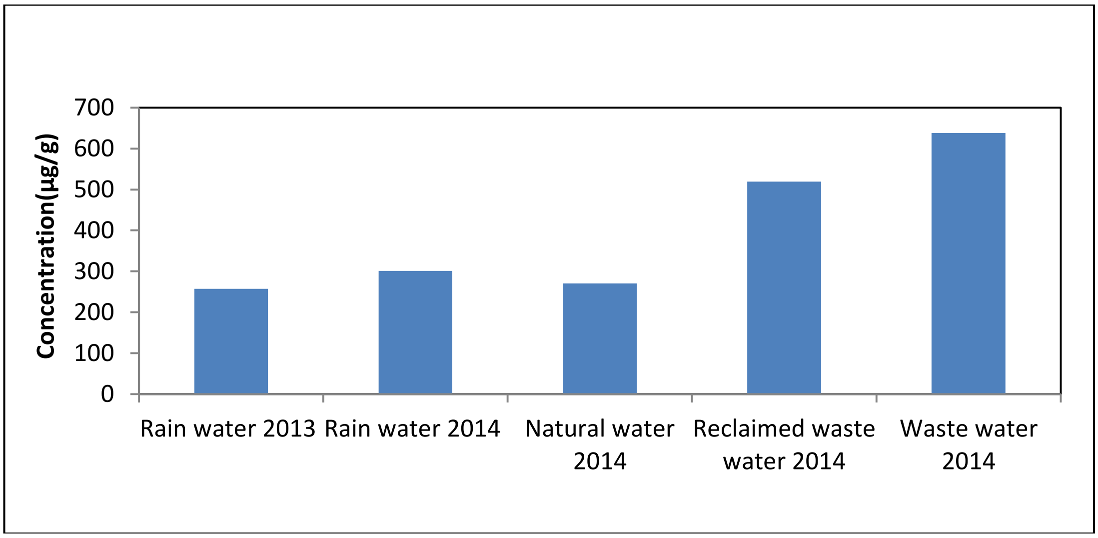


Fig.(4.19): Concentration of Magnesium in olive oils for all sampling sites according to their irrigation system of crops of 2013 and 2014

The Fig.(4.19) shows that the highest concentration of Mg for the samples that had trees that were irrigated by waste water and reclaimed waste water in the range between 257.233 up to 637.9324 µg/g, which is greater than the concentration of previous studies according to table (1.1).

4.6.5 Iron (Fe)

Iron is an essential metals for humans body, because it contains number of protein, enzymes and having hemoglobin and transports oxygen through blood for all tissues in human body (Washington, 2001). Each 450 – 500 ml of human blood contents 200 – 250 mg of iron.

The maximum concentration for iron is 100 mg per day. If iron is taken at low concentration it can cause anemia disease while the high concentration can damage tissues (Nancy *et al*, 1999). Iron increases the absorption of Cd, Pb, Al (Goyer, 1997). The concentrations of iron according to their irrigation system are shown in Fig. (4.20).

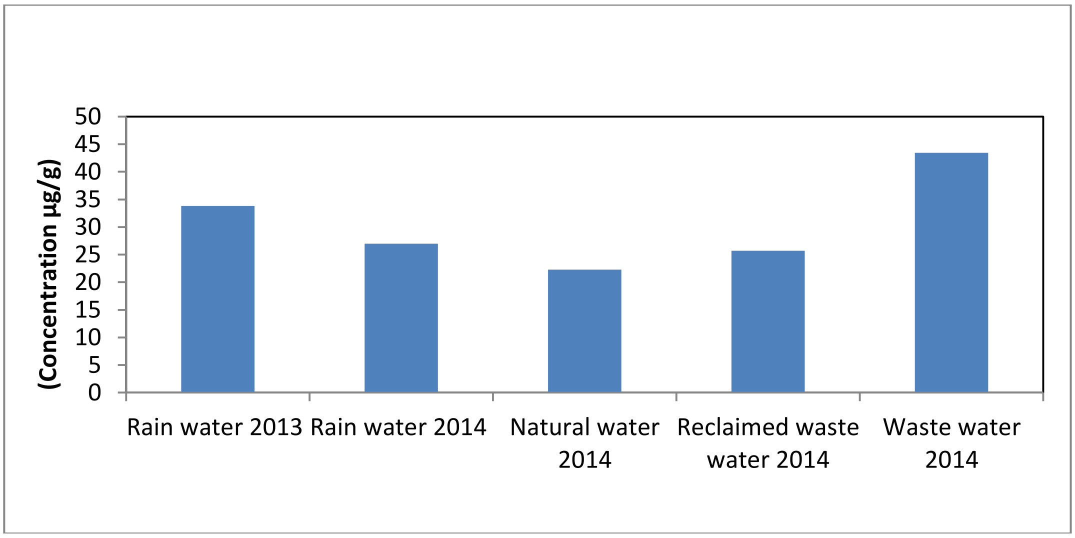


Fig.(4.20): Concentration of Iron in olive oils for all sampling sites of crops of 2013 and 2014

Fig. (4.20) shows the maximum value for iron metal in the measured concentration is 43.449 µg/g for samples which their trees were irrigated by waste water of crop 2014, and this measured value is greater than the standard value 3 µg/g for IOOC (IOC, 2015).

4.6.6 Cadmium (Cd)

Cadmium is a very toxic trace metal which its spread through blood, and interacts with zinc (Lyaud *et al*, 2012;). The concentrations of cadmium according to their irrigation system are shown in Fig. (4.21).

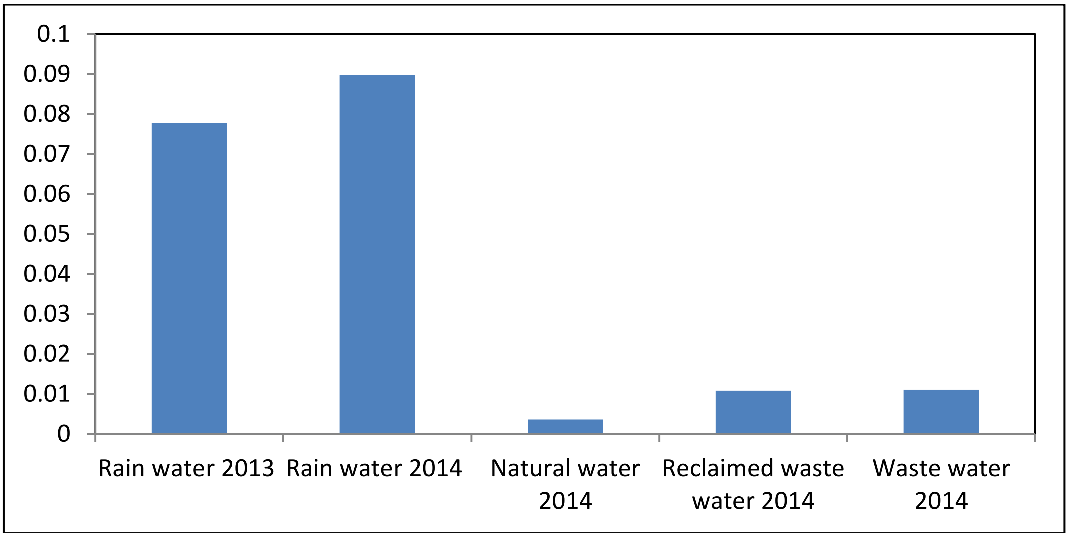


Fig.(4.21): Concentration of Cadmium in olive oils for all sampling sites of crops of 2013 and 2014.

The highest Cd concentration in olive oil sample was 0.0898 µg/g where the lowest value was 0.0036 µg/g for sample S_2 with trees irrigated by natural water as shown in Fig.(4.21). The measured values of Cd concentration in all olive oil samples lower than standard value according to table (1.1).

Cadmium particles released into air because of its use in various industries and emitted from car exhaust gases, where Hawara town is considered as an industrial area and heavily traffic area. These reasons gives highly concentration of cadmium for sample S_2.

4.6.7 Copper (Cu)

Copper is one the main important elements for human body which plays an essential rule for producing enzymes and is essential for biological activities (Hashmi et al, 2005). Cu is a very toxic If it is taken at high concentration it can causes many problems for human such as brain damage (Bonham *et al*, 2002: Zhu *et al* , 2011). The concentrations of copper according to their irrigation system, which they are shown in Fig. (4.22).

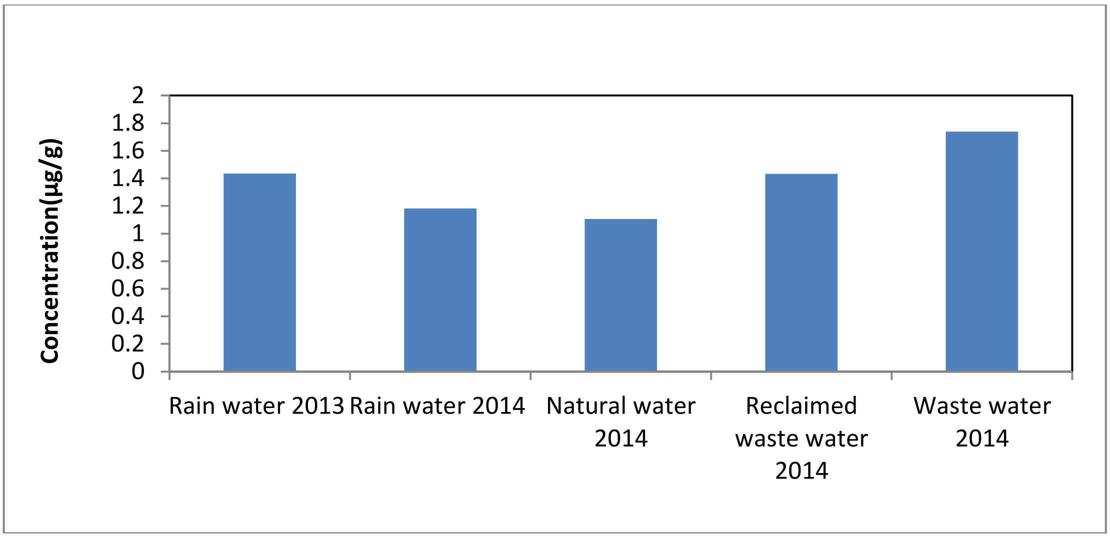


Fig.(4.22): Concentration of Copper in olive oils for all sampling sites of crops of 2013 and 2014

The range of copper is 1.183 - 1.740 µg/g. The measured values are greater than 0.1µg/g for IOOC standard (IOC, 2015). Samples with trees depend on natural water had the lowest copper concentration, but sample S_4 with trees irrigated by wastewater had the highest concentration.

4.6.8 Manganese (Mn)

Manganese metal is essential for humans at low concentration, but if the intake is at a high concentration, it can cause many problems for the human body. The concentrations of manganese according to their irrigation system are shown in Fig. (4.23).

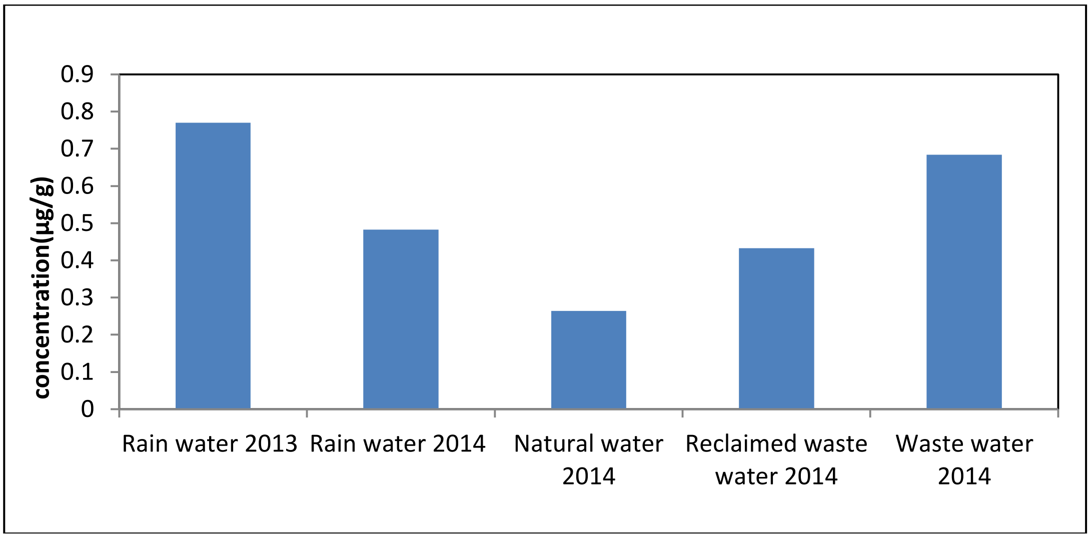


Fig.(4.23): Concentration of Manganese in olive oils for all sampling sites of crops of 2013 and 2014

The minimum and maximum levels of Mn were 0.2644 and 0.7702 µg/g for samples S_2 and S_{1a} with trees were irrigated natural water and trees depend on rain water respectively. The measured values for samples S_2 in the range of standard value while the concentration of sample S_{1a} are greater than the standard value.

4.6.9 Zinc (Zn)

Zinc is one of the main essential trace element in the body which needs at low concentration for growth and considered as cofactor for many enzymes (Ashraf, 2014; Maggini *et al*, 2010). The concentrations of zinc according to their irrigation system are shown in Fig. (4.24).

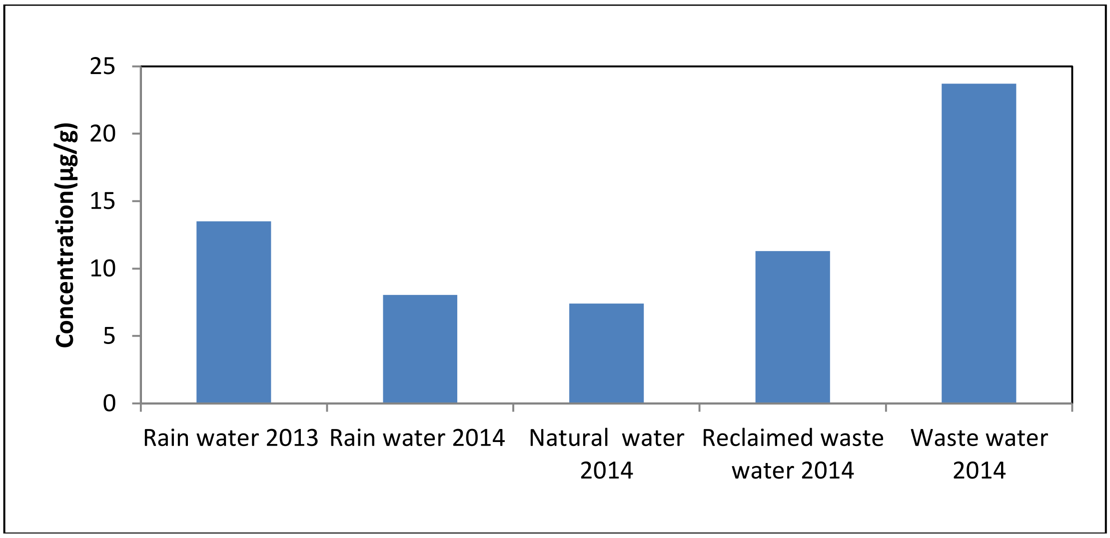


Fig.(4.22): Concentration of Zinc in olive oils for all sampling sites of crops of 2013 and 2014

The zinc content for the measured samples ranged from 7.4032 to 23.7126 µg/g. The sample with trees irrigated with natural water had the lowest value, while the samples which their trees irrigated by waste water had the highest value for zinc concentration. The concentration of Zn in all measured samples were higher than standard values.

Chapter Five

Conclusion

The results for the physical properties and some metals of olive oil samples for different irrigation system are given in table (5.1)

Table (5.1): The measured density and viscosity at 20℃, acidity and the concentration of some metals for different irrigation system of crops 2013 and 2014

Region	Type of Irrigation	Sample	Crops	Mass Density (gm/cm^3)	Viscosity (cP)	%FFA	Cd	Mn
Misseli and Hawara	Rain water	S_{1a}	2013	0.9161	70.2	2.34	0.0778	0.7702
Misseli and Hawara	Rain water	S_{1b}	2014	0.9169	69.5	1.85	0.0898	0.4830
Shillo	Natural water	S_2	2014	0.9179	72.3	2.73	0.0036	0.2644
Anabta	Reclaimed waste water	S_3	2014	0.9198	73.2	3.03	0.0108	0.4324
Deir Sharaf and Brouqen	Waste water	S_4	2014	0.9205	80.4	3.35	0.0110	0.6836

The measured density of olive oil samples of crop 2014 has the highest value. It was found to be 0.9205 gm/cm^3 with standerd deviation less than 2% for irrigated samples by waste water because the concentration of some metals shown to be more than the other samples. The measured results shown that the nature of irrigation affects the density results.

The experimental results of viscosity of olive oil decreased as a function of temperature. The viscosity of olive oil samples differ from one location to another according to their irrigation system. The results of viscosity values of 2014 crop irrigated sample by waste water are greater than the values of olive oil viscosities for other locations. The viscosity of olive oil with trees irrigated by reclaimed waste water and pure water of crop 2014 are also greater than viscosity for samples depend on rain water of crops 2013 and 2014. This difference may be due to concentrations of metals in waste water. The standerd deviation for different samples is 3%.

The measured acidity results of olive oil increased for samples with trees irrigated by waste water and classified to Lampante oil. The overall results in this study indicate that the acidity in the analyzed olive oil samples also increased for samples irrigated by reclaimed waste water and natural water are greater than samples depends on rain water. The storage period effects the oxidation of olive oil samples.

The samples were irrigated by waste water contain higher amount of calcium, sodium, potassium, magnesium and zinc. The concentration of iron, copper and lead for irrigated samples by waste water are greater than concentration of established by standards of IOOC. Waste water have high concentration of calcium, sodium, potassium, magnesium, zinc, copper, iron and lead, so that for this reason these metals are found in high concentration for samples irrigated by waste water. The concentration of cadmium and manganese found at high concentration for Hawara samples because it is industrial area.

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Appendix A

The densities as a function of temperature for some samples are shown in Figs. (A.1 – A.4)

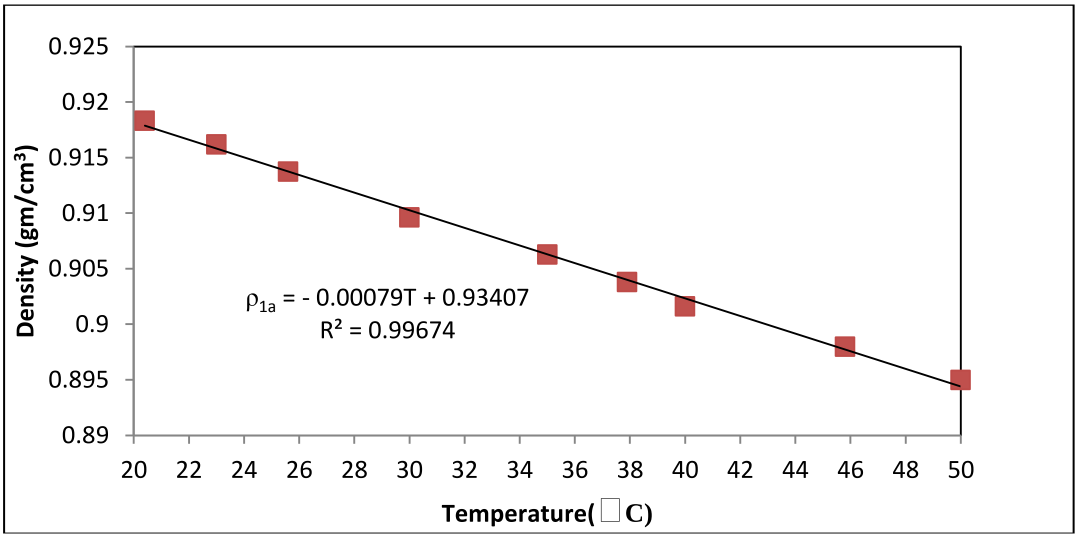

Fig.(A.1): The measured density for sample S_{1a} in with trees depend on rain water, form Hawara town of crop 2013

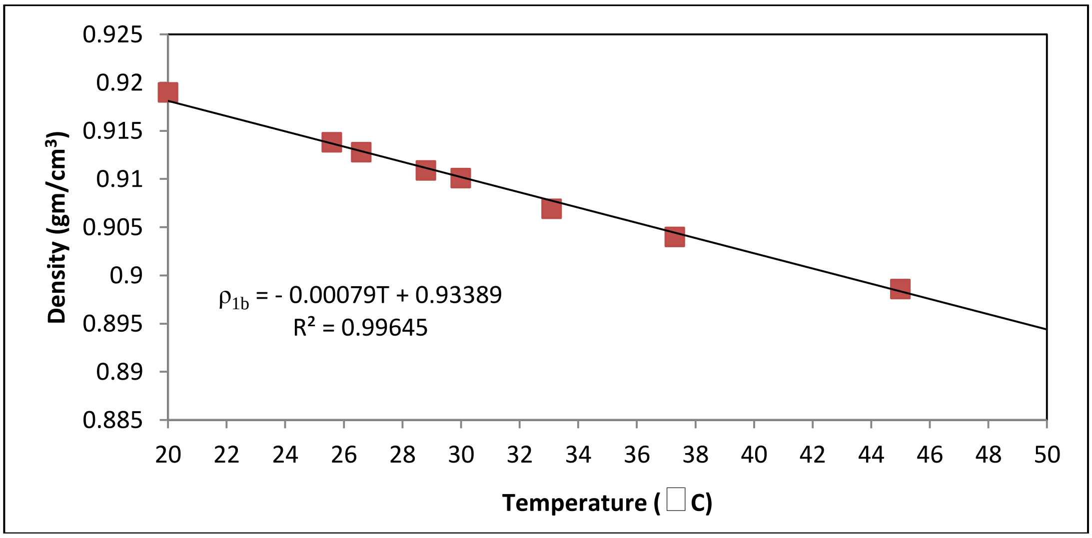

Fig.(A.1): The measured density for sample $S_{1b}$with trees depend on rain water, form Hawara town of crop 2014

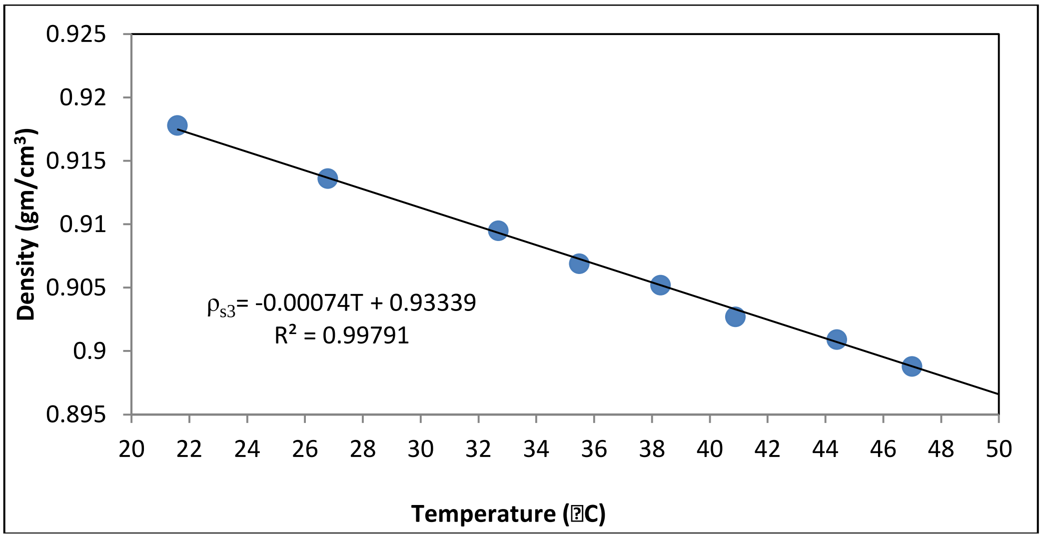

Fig. (A.3): The density measured of sample (S_3) with trees irrigated by reclaimed waste water at different temperature from Anabta of crop 2014

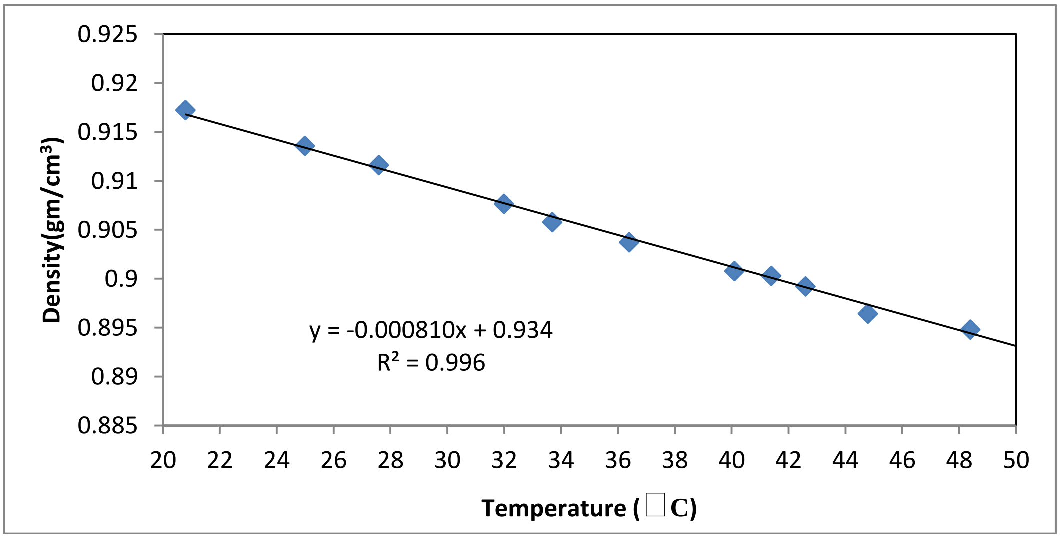

Fig. (A.3): The density measured of sample (S_4) with trees irrigated by waste water at different temperature from Deir Sharaf of crop 2014

Appendix B

The viscosities as a function of temperature for some samples are shown in Figs. (B.1 – B.4)

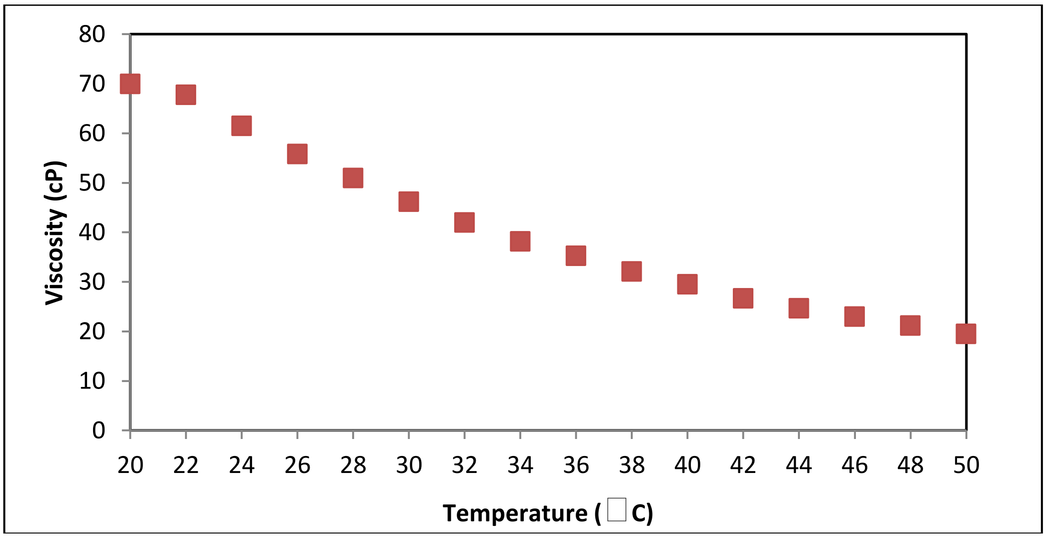

Fig.(B.1): The measured viscosity for sample S_{1b} with trees depend on rain water, form Hawara town of crop 2014

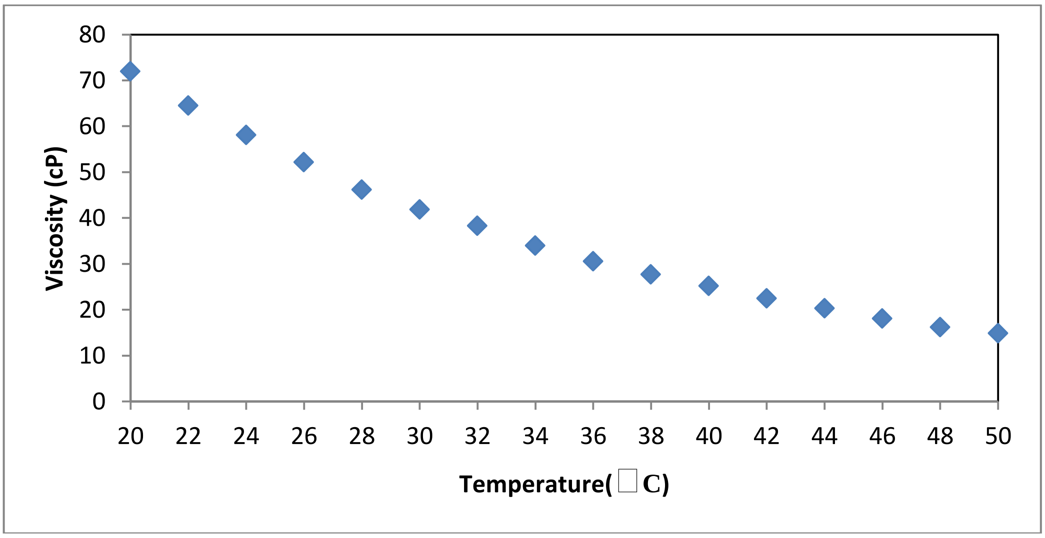

Fig.(B.2): The measured viscosity for sample S_{1a} with trees depend on rain water, form Hawara town of crop 2013

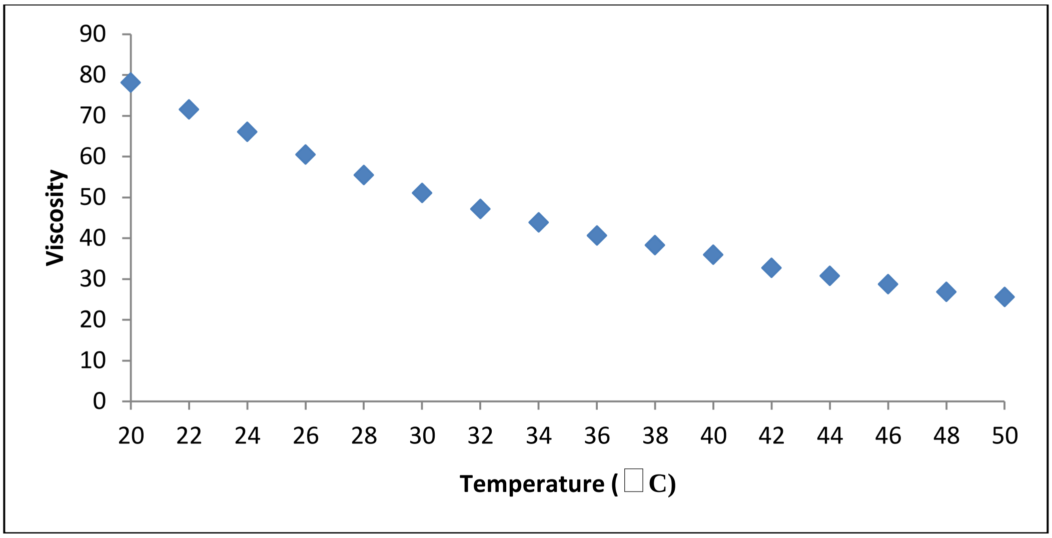

Fig.(B.3): The measured viscosity for sample S_3 with trees irrigated by reclaimed waste water, form Anabta of crop 2014

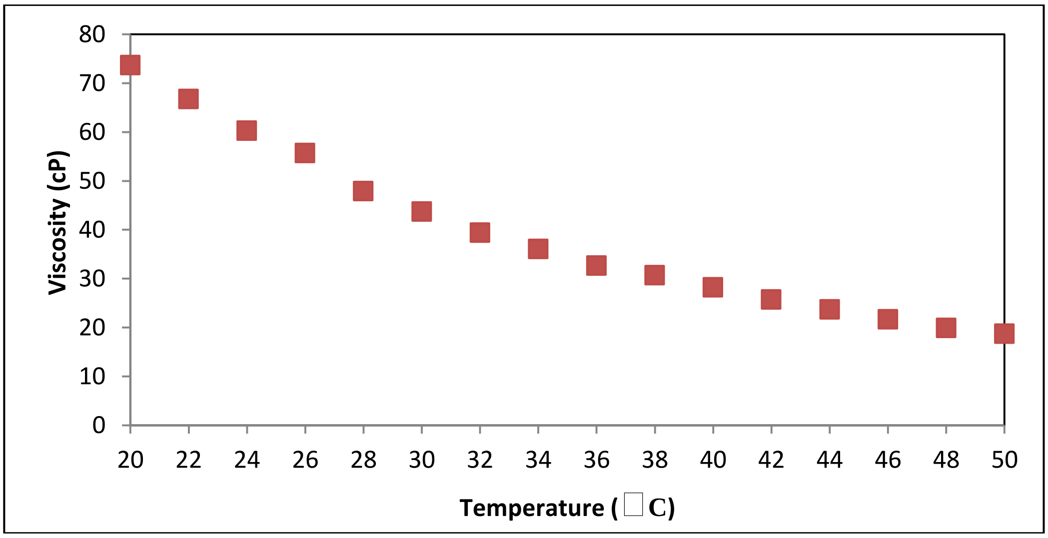

Fig.(B.4): The measured viscosity for sample S_4 with trees irrigated by waste water, form Deir Sharaf of crop 2014

جامعة النجاح الوطنية
كلية الدراسات العليا

تأثير المياه العادمة المستخدمة في الري على بعض الخصائص الفيزيائية لزيت الزيتون في فلسطين

إعداد
لينا حسن عودة

إشراف
ا. د. عصام راشد عبد الرازق
ا. د. شريف محمد مسامح

قدمت هذه الأطروحة استكمالا لمتطلبات الحصول على درجة الماجستير في الفيزياء بكلية الدراسات العليا في جامعة النجاح الوطنية في نابلس، فلسطين.

2015

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أ. د. شريف محمد مسامح

الملخص

تناولت هذه الدراسة عينات من زيت الزيتون مختلفة في أنظمة الري من مناطق مختلفة في فلسطين. حيث تم قياس الكثافة واللزوجة والحموضة ومعامل الانكسار وأظهرت النتائج العامة زيادة الحموضة لعينات سقيت أشجارها بالمياه العادمة وان معظم العينات صنفت بأنها زيت زيتون بكر. ومن ناحية أخرى أظهرت دراسة اللزوجة والكثافة لعينات زيت الزيتون لمحاصيل 2014 من مناطق مختلفة أن اللزوجة تتأثر بطبيعة الري وتزداد قيمتها للعينات المروية بالمياه العادمة وكذلك تم مقارنة نتائجنا لتركيز المعادن مع الدراسات السابقة ووجد أن تركيز العناصر في العينات المروية بالمياه العادمة أكثر من غيرها من العينات للدراسات السابقة